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Abstract Selected activities of the Materials Department at Risø National Laboratory during 1990 are described. The work is presented in three chapters: *Materials Science*, *Materials Engineering* and *Materials Technology*. A survey is given of the Department's participation in international collaboration and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists, publications, lectures and poster presentations are included.

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1 Introduction

Research Strategy

Research and development activities must meet a variety of demands at the same time. Firstly, the research must be of high quality and it must be visible. Secondly, research and development activities must serve the needs of society and must meet the specific demands of the industrial sector. Finally, national work must be an integral part of the international effort. These guidelines are followed at Risø National Laboratory where medium and long term strategic research is carried out within the areas of energy, environment and materials. The work in the Materials Department* concentrates on materials issues. For a number of programmes the energy aspect is also an integral and important part of the activities. Examples of such programmes are solid oxide fuel cells, divertor materials for fusion reactors and rotor blades for wind mills.

Materials research and development in the Materials Department follows the guidelines mentioned above in the way that our applied programmes are based on long range research within fundamental areas. The fundamental programmes extend our knowledge of materials, for example through modelling, characterization of microstructure and texture and irradiation defects, whereas applied programmes concentrate on areas such as engineering, materials testing and materials performance. Finally, materials technology, involving processing and manufacturing, forms the necessary basis for almost all of the work on advanced materials. The groups of materials studied are metals, ceramics and polymers as well as mixtures of such materials in the form of composites.

The work in the department is organized in the form of programmes and projects, which are divided into three main areas or disciplines, namely materials science, materials engineering and materials technology. The different areas, programmes and projects are given in this table.

MATERIALS SCIENCE
Modelling, structure and properties
Composite materials
Polymers, structure and properties
Irradiation defects and fusion materials
Modelling and characterization,

MATERIALS ENGINEERING
Design and testing
Structural mechanics
Materials testing
Non-destructive evaluation
Materials performance

MATERIALS TECHNOLOGY
Fabrication and processing
Powder metallurgy
Polymer composites
Engineering ceramics
Joining of materials
Component manufacturing
Solid oxide fuel cells

Area programmes and projects

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*Risø National Laboratory was reorganized in 1990 and the Metallurgy Department changed its name to the Materials Department. The activities of the Department were not modified except within the activities on polymer materials which were expanded through the transfer of the polymer group from the Chemistry Department.

Resources

The present staff situation, in which the Department has been able to maintain controlled growth by hiring young engineers and scientists, is satisfactory. This expansion is necessary to maintain a favourable age distribution and to ensure a constant inflow of up-to-date knowledge as acquired by candidates and Ph.D's from Danish and foreign universities.

Developments in financial resources are at present less satisfactory. In 1990, Risø and the Department faced severe cuts in basic funding. The Department has been able to offset these cuts by increasing the amount of project funding supplied by various sources outside Risø. This change in the income structure points towards a future with less money for basic research and reduced investment in advanced research equipment. The problem is exacerbated due to a large part of the project funding not covering all expenses; such projects must be supplemented by basic funds. As an example, the EFC normally covers only 50% of the total expenses on a contract. In the present economical climate in Denmark, an increase in basic funding is not to be expected in the near future. Therefore the Department must look for sources of income which more fully cover individual project research expenses. One possibility is increased contract work for third parties. In addition, the development and supply of materials, process technology and advanced products on a pilot plant scale must be looked into.

Research

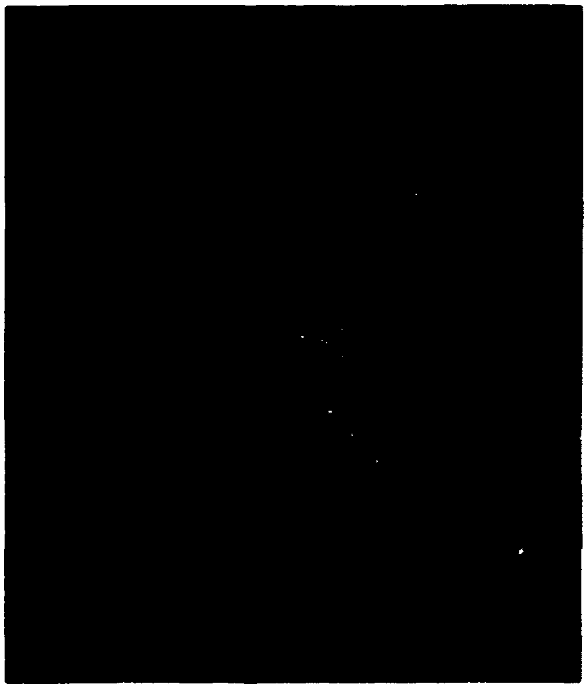
In this report the work in the Department is described according to the various areas given in the table above. Research projects may also be related to energy and industry. This relationship is described in the following and is supplemented with a subsection on basic research.

Energy Related Research

The primary materials research activities in this area are related to the development of fuel cells. Fuel cells have a high potential for the production of electricity and heat with high efficiency and low atmospheric pollution; hence the high priority given to it by the Ministry of Energy. The work focuses on materials development for Solid Oxide Fuel Cells (SOFC) with the aim of constructing prototype fuel cells. A national programme has been initiated for the period 1990-92 with the participation of the utilities, industry, research institutes and the Ministry of Energy. The management of the whole programme is placed in the Department. Energy related research in the Department also encompasses the design, materials development and materials testing for wind mills which have a high priority within the Danish energy programme. Research is also continued, although on a limited scale, on hydrogen storage in metals.

The research within the nuclear fission area has now been terminated, following the completion, in 1990, of the Third Risø Fission Gas Project. This marks the end of about 30 years of conventional nuclear research. In consequence a decommissioning of the Hot Cell facility has been initiated. This work is planned for the period 1990-1993 and it is foreseen that the space which will become available will be used for other purposes, e.g. laboratories for fuel cells development and for polymer research. Finally within energy research, the Department participates with materials research in the European fusion technology programme.

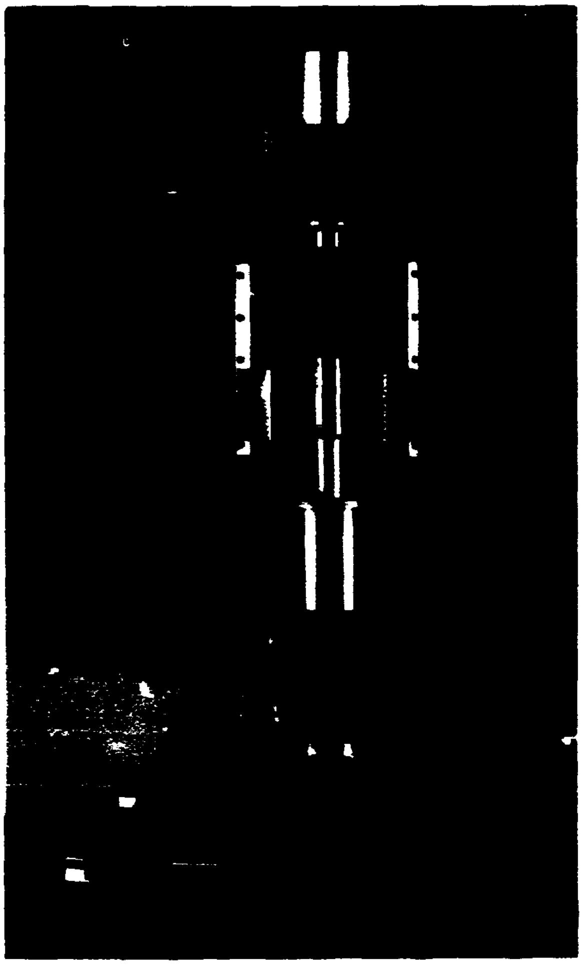
A prototype solid fuel cell being tested in a furnace at 1000°C with supply of hydrogen and air to the electrodes.



Industry Related Research

Within the general area of materials research the Department is an active partner in the Danish Programme on Materials Technology. This programme has a budget of DKK 495 mio. over the period 1989-1994. About half of this money will be spent on financing five »centres« which are formalized collaborative arrangements between partners from industry, research organizations and universities. The research programmes for each centre is agreed upon amongst the partners; the tasks are primarily those given a high priority by the industrial partners. The Department participates in three centres, namely the centre of polymer composites, the centre of powder metallurgy and the centre of engineering ceramics. The administration and coordination of two of the centres is placed in the Department. The work in the centres is supplemented by »programmes« with fewer participants but still with the objective that the results must be of industrial interest. About one third of the total budget is reserved for such programmes where the Department participates in two: one on polymers and another on modelling mechanical properties of materials. The administration and coordination of these programmes is placed in the Department. In total, the annual income to the Department from this national programme is about DKK 10 mio. per year. Our large involvement in the national Programme on Materials Technology reflects the strong position held by the Department within Danish materials research and development. It also illustrates how important this programme is for the development of the Department's expertise within the materials area.

Industry related research in the Department also includes a number of individual projects in most cases carried out on a proprietary basis. Such work relates to design, materials selection and materials testing, non-destructive evaluation and failure analysis. Furthermore the Department undertakes work as an industrial subcontractor in areas where special expertise has been built up, e.g. in brazing of components for the electronics industry. Finally the Department operates a fuel element plant for the manufacturing of low enriched fuel elements for research reactors.



Newly constructed test rig for in-situ neutron diffraction. Internal stresses in metal matrix composites will be determined during loading at elevated temperatures.

Basic Research

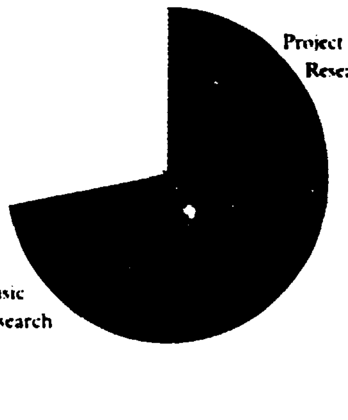
The long range basic programme covers areas related to the applied programmes described above. Even though the research is of fundamental nature, projects are initiated in response to specific technological and engineering demands for new and improved materials. Within this framework, research topics to be tackled are selected on scientific merit. The research covers modelling of materials, behaviour and characterization of structure and properties. The materials investigated are metals, polymers and ceramics. Composites research is a special area and materials are here metal-matrix composites, polymer composites and ceramic matrix composites. In the study of composites behaviour, a major activity is the use of neutron diffraction to measure internal elastic strains in order to study thermal and mechanical stresses. Such stresses build up both during manufacturing and during the exposure to loads and temperatures when the composites are used in engineering components.

Polymer research is newly integrated into the Department and the general objective is to explore the relationship between properties and the molecular and microstructure of polymers and mixtures of polymers. An important part of this work is to study and modify surfaces of various polymeric systems.

Radiation damage of metallic materials is also studied especially the problem of simulating the damage which will take place in a fusion reactor. The experience gained within this area on the formation, transport and accumulation of crystal defects and gases is now being implemented in studies of defects in ceramic materials and of the behaviour of gases in materials.

An important part of the basic research programme is the development of advanced experimental techniques within the areas of neutron diffraction (especially texture and internal stress measurement), small angle neutron scattering, positron annihilation and electron microscopy. A part of the work is the development of fully automated techniques for the analysis of experimental outputs, e.g. in the form of micrographs and diffraction diagrams.

Distribution of manpower on research supported by basic and project funds.



International Cooperation

Within nearly all research areas of the Department there is an active international cooperation. This is reflected in the many foreign students and guest scientists who have worked in the Department during the year. This cooperation is also demonstrated by the many joint authorships of the scientific publications of the Department.

Participation in Danish research programmes has led to an active engagement in many programmes under the auspices of the EEC. Of special importance is the BRITE/EURAM programme. The Department participates in research related to materials modelling, composites, materials testing, engineering ceramics, powder metallurgy, brazing and materials for fuel cells. Our activities in nine BRITE/EURAM programmes are supplemented by participation in two Joule Programmes on fuel cells and on the design and testing of rotor blades for wind mills.

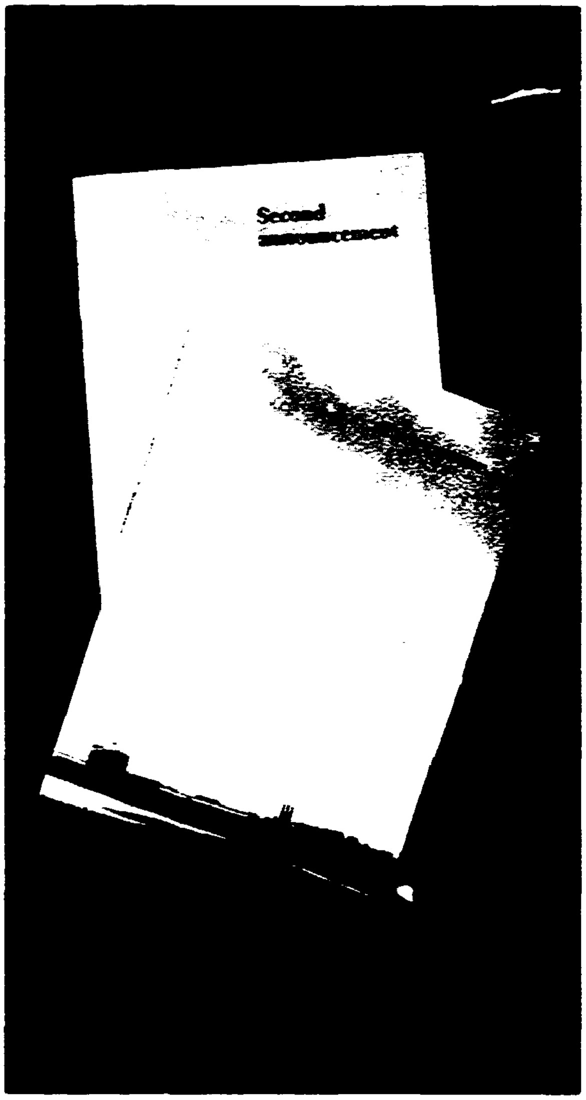
The broad international collaboration as well as the close relationships between the programmes of the Department within the European and the Danish framework support an important role as a »bridge« between international and national materials research. This function has an important bearing on issues such as technology import and technology transfer. A new initiative in this area is to actively engage the Department in the formulation and organization of new international projects with the special objective of increasing the involvement of the Danish industry in European sponsored research.

As part of the international collaboration, staff members are representated in many committees, advisory groups etc.

Achievements

A number of scientific and technical achievements made during the year can be mentioned. The Third Risø Fission Gas Project has been successfully completed and a national programme on solid oxide fuel cells has been initiated. In the area of materials modelling, two large contracts were obtained: one within the Danish Programme on Materials Technology, »Modelling of the Mechanical Behaviour of Materials« and one within EURAM/BRITE on Modelling of Hot Deformation of Aluminium Alloys. These projects combine theoretical modelling with experimental characterization of texture by neutron diffraction and of lattice misorientations by electron back scattering patterns. This latter technique has successfully been automated. In the area of composites, the behaviour of short fiber metal matrix composites has been investigated both theoretically and experimentally. A publication of this work together with the Department of Metallurgy and Materials Science at Cambridge University was awarded the Acta Metallurgica Outstanding Paper Award for 1989.

In the technology field it was reported in last years progress report that an ultra light polymer composite racing canoe was designed and built in collaboration with students from the Engineering Academy. In this canoe one of the students won the World championship in marathon canoeing. Within the field of polymers a process for surface modification has been patented and this invention has created very positive interest both from the scientific and from the industrial community. Within education a new venture has been the participation in the preparation of a comprehensive national course for technicians and engineers on materials.



Symposia and Workshops

An important activity in the Department during 1990 has been the organization of the »Eleventh Risø International Symposium on Metallurgy and Materials Science«. This symposium followed the format of the Risø International Symposia and was arranged in collaboration with the Danish Center of Engineering Ceramics. The number of participants was about 130. The subject for 1991 has been chosen to be »Metal Matrix Composites. Processing, Microstructure and Properties«. This symposium is co-sponsored by the Danish Centre for Powder Metallurgy. Another activity has been a summer school arranged by the Nordic Committee for Thermal Analysis. The duration of this school was three days and the number of participants was about 100. Finally the Department was host to the annual meeting of the Commission of European Communities Working Group »Hot Laboratories and Remote Handling« and of the Expert Group on Structural Materials within the European Fusion Programme.

Publications and Reports

The results of the scientific and technical work are primarily reported in international scientific journals and conference proceedings. However, a relatively big part of the contract work appears in reports of limited circulation and in confidential reports. In order to ensure a broad circulation of the results of the research, a number of overview papers have been written in Danish and circulated widely to interested parties.

Participation in the technical exhibition »Tech Trans '90« held in Herning, Denmark.

Public Relations

The public relations activities mentioned in the sections on Visits and Publications and Reports have been supplemented by new initiatives. Firstly the Department participated in a major technical exhibition »Tech Trans '90« in Herning. The Department had its own stand and participated also in the stand of the Centres under the Danish Programme on Materials Technology. Also to be mentioned is the arrangement of an exhibition in combination with the annual meeting of the Society of Plastic Industries in Denmark (the Division for Fibre Reinforced Polymers) in Kolding. Finally the Department arranged a seminar for journalists and editors, who cover technical and research subjects in Danish newspapers and technical journals. The subjects presented were contained within the main theme of prospects for advanced materials in modern society.

Visits

The Department was host to many guest scientists from around the world. Of these, a total of 17 (listed in Chapter 9) stayed for extended periods to work on projects in collaboration with staff members.

Education

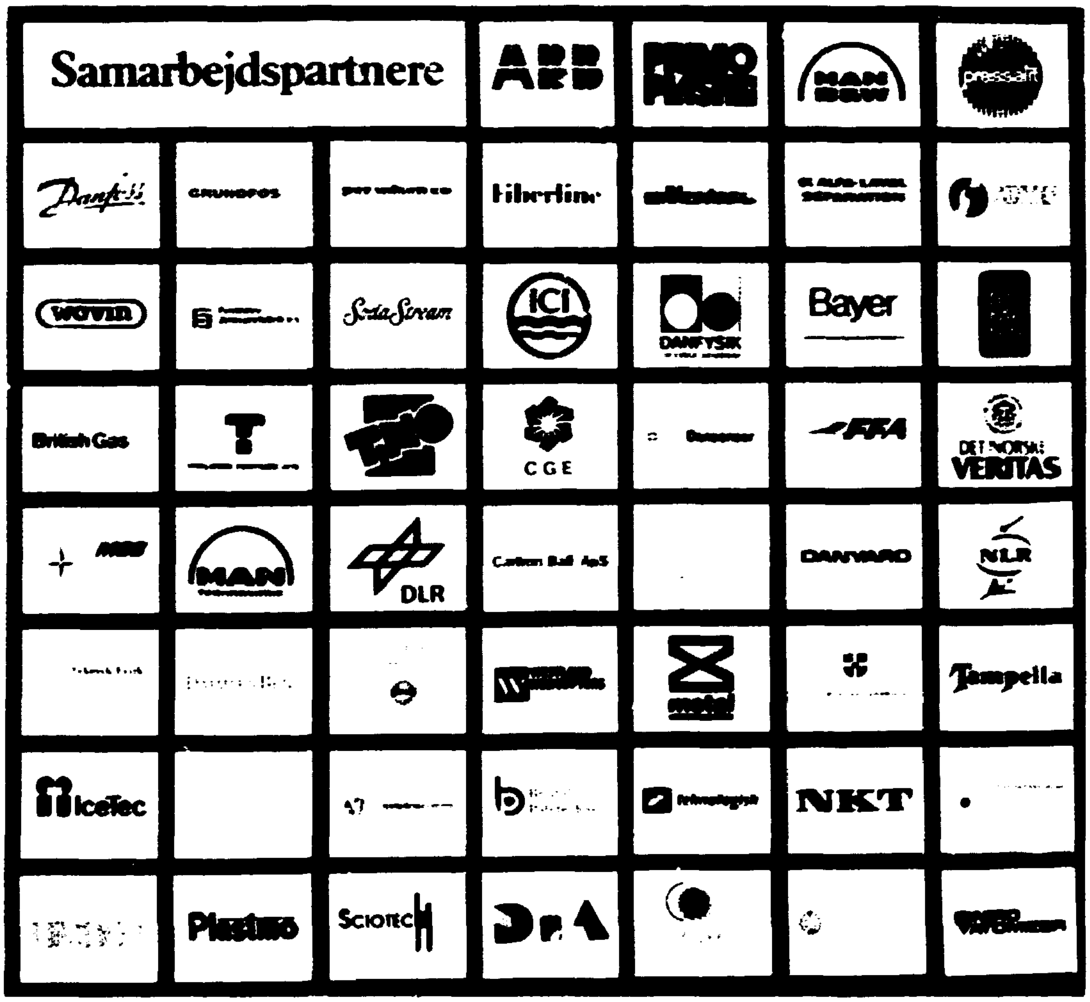
Education and training are an important activity of the Department with many of the staff members serving as external lecturers and examiners at university undergraduate and post-graduate levels. Also, undergraduates and graduates from engineering schools in Denmark and abroad have carried out experimental work and received academic training in the Department. in 1990, 9 undergraduates and 7 post-graduates were supervised by Department staff members. Several post-graduates have also studied in the Department. Besides this academic training the Department also teaches a number of apprentices in mechanics and electronics.

At present, educational activities are being increased and extended by participation in the planning and execution of a comprehensive course for technicians and engineers on materials properties, processing, testing and product design. This course is under the sponsorship of the Danish Ministry of Education (law no. 271) and is carried out as a collaboration between a number of research institutes, universities and technical schools in Denmark. The Department's effort will be concentrated on advanced materials especially properties, design and technology in the area of polymers, polymer composites and sandwich materials. The Department coordinates this part of the course and participates also in other parts such as engineering ceramics and powder metallurgy.

Concluding Remarks

During 1990 the Department was able to maintain its rather firm scientific, technological and economic basis on which to build its activities in the coming years. The basis of the Department has in some areas been strengthened significantly in 1990, an example is the very promising national cooperation between the utilities, industry, research institutes and the Ministry on research related to energy issues. Another example is the positive development in the collaboration with Danish industry within materials technology and the beginning of an integration of such work into international research programmes in which the Department is involved. Finally a large national initiative on materials education in which the Department takes an active part will ensure that the research results obtained in Denmark and elsewhere will be widely disseminated in Danish society.

A poster from the Herning exhibition showing many of the Department's industrial and academic research partners.



2 Materials Science – Modelling and Characterization

Efforts to improve the inherent properties of materials are based on our ability to characterize, understand and finally to modify microstructure. The research in this area, although of a fundamental nature, is often initiated in response to specific technological and engineering demands for new and improved materials. The research themes in Materials Science are therefore closely related to the applied programmes within the Department. Much of this basic research is carried out in close collaboration with colleagues from universities and government research laboratories around the world.

Current research activities are concentrated in the following areas:

- Quantitative modelling of deformation microstructures - texture and recrystallization.
- Mechanical properties and modelling related to the behaviour of single crystals and polycrystalline metals as well as metal matrix composites, dispersion strengthened materials and polymer matrix composites.
- Defects and microstructure caused by irradiation with energetic particles. Mechanical properties of materials for fusion reactors.
- Ion conductivity in solid oxide electrolytes for use in fuel cells.
- Development of advanced techniques for the characterization of materials. Neutron diffraction and small angle neutron scattering, electron microscopy and positron annihilation.
- Polymer chemistry and physics.

2.1 Models for the Mechanical Behaviour of Single-Phase Materials

The mechanical behaviour of materials may be described in »constitutive models« which express the relation between stress and strain. The formulation of such constitutive models is one of the ultimate goals of materials science. The constitutive behaviour is determined by the structure, mainly the microstructure and the texture, and therefore modelling of the microstructural and the textural evolution is a logical step towards the final goal of constitutive models. Such a model has been formulated for aluminium.

A unified view of the stages of structural evolution in monotonic and cyclic plasticity was recently obtained by drawing »fatigue mechanism maps« in plastic strain/cycle number space. Such maps were developed for iron-silicon alloys on the basis of models and available observations.

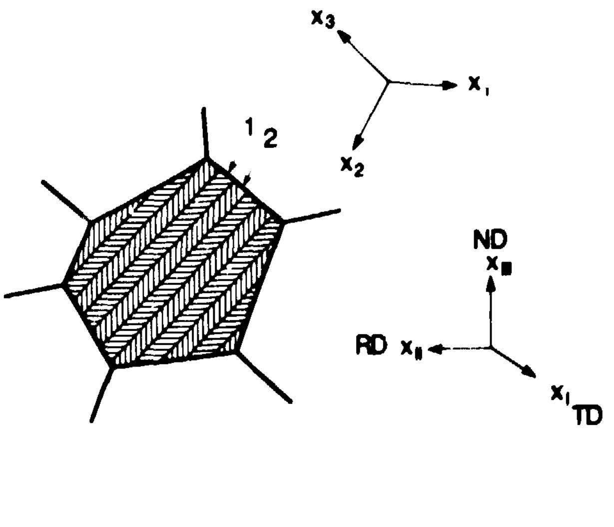
Grain Subdivision in Aluminium

In rolled aluminium the grains are subdivided in »cell blocks« as sketched in the figure opposite. There seems to be two families of cell blocks, each with a specific combination of active slip systems. It has been suggested that the number of active slip systems in the cell blocks is less than the 5 required by the Taylor model. The cell blocks are separated by dense dislocation walls or first generation microbands, DDWs/MBs. The DDWs/MBs are approximately parallel to the transverse direction (which is perpendicular to the plane of the paper in the sketch), making angles of about 40° with the rolling direction (with a considerable spread). At the later stages of deformation another set of intersecting DDWs/MBs forms in some grains.

A simple model has been formulated for the situation with only one system of DDWs/MBs. The model allows strain differences between the two families of cell blocks, but strain continuity is maintained between the cell blocks (across the DDWs/MBs). The grain as a whole (i.e. the two families of cell blocks taken together) follows the macroscopic strain. It turns out that there are solutions with an average of less than 5 active slip systems in each cell block: 5 in one family and 3 in the other or 4 in both families. The total of 8 slip systems does not necessarily mean 8 different systems: the same systems may be active in both families. For a given grain (with a given orientation) there are about 160000 solutions with a total of 8 active slip systems. As is the case for the normal Taylor model, one selects the minimum-shear solutions. In most cases one finds a large number of solutions, i.e. there is a high degree of ambiguity.

The resulting active slip systems are always among the 6 or 8 systems which may be active when the normal Taylor model is used on the grain considered. Furthermore, the average M factor for the two families of cell blocks is always identical to the Taylor M factor for the grain considered. It is by no means certain that the present model provides a correct description of the slip pattern in the grains subdivided in cell blocks, but the model shows that one may provide a mathematically consistent description of the subdivision in cell blocks with less than 5 active slip systems.

Sketch of grain sub-division into two families of cell blocks during deformation by rolling.



$$\varepsilon(1)_{11} = \varepsilon(2)_{11}$$

$$\varepsilon(1)_{22} = \varepsilon(2)_{22}$$

$$\varepsilon(1)_{12} = \varepsilon(2)_{12}$$

$$\varepsilon(1)_{\mathrm{I\,I}} + \varepsilon(2)_{\mathrm{I\,I}} = 0$$

$$\varepsilon(1)_{\mathrm{II\,II}} + \varepsilon(2)_{\mathrm{II\,II}} = 2E$$

$$\varepsilon(1)_{\mathrm{I\,II}} + \varepsilon(2)_{\mathrm{I\,II}} = 0$$

$$\varepsilon(1)_{\mathrm{II\,III}} + \varepsilon(2)_{\mathrm{II\,III}} = 0$$

$$\varepsilon(1)_{\mathrm{III\,I}} + \varepsilon(2)_{\mathrm{III\,I}} = 0$$

The eight equations for strain continuity during deformation of the grain shown in the sketch below.

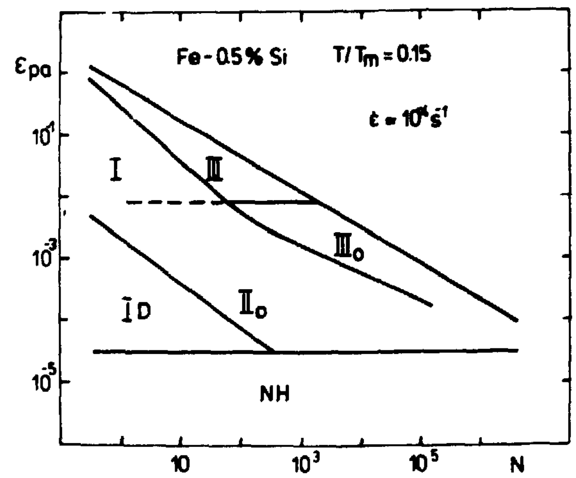
Mapping of Cyclic Plasticity in bcc Alloys

Mechanism maps for work hardening and fatigue in metals were recently introduced for pure and well-annealed face-centred cubic (fcc) metals in diagrams of plastic strain versus cycle number. The successive stages of structural (and textural) evolution and cracking appear as two-dimensional fields, whose sizes and positions were partially predicted in terms of current physical models.

It is of interest to know the effect of crystal structure and strengthening mechanisms on the field pattern in the maps. Is is clear that maps for the body-centred cubic (bcc) metals and alloys must be qualitatively different to those for the fcc metals. Essentially, the bcc materials display a ductile to brittle transition when the temperature is reduced and/or the deformation rate is increased. The effect is apparently caused by the three-fold dissociation of the bcc screw dislocation core, which entails a strong lattice friction at low temperatures.

The construction of the maps relies heavily on the availability of a systematic and fairly comprehensive set of experimental observations. Such a data set is now becoming available for the iron-silicon system, and an attempt (in collaboration with Institute of Physics, Czechoslovak Academy of Sciences, Prague) was made to sketch maps for iron-silicon single crystals on the basis of the existing data. A conspicuous novel feature of these maps is the appearance of a low-amplitude field labelled NH (»no hardening«). Field NH is characterized by low dislocation densities and negligible cyclic hardening, reflecting a deformation mode in which the plastic strain is carried mainly by mobile edges. The effect of adding silicon is to decrease the edge mobility, thereby reducing the difference between screw and edge mobilities and hence reducing the extent of field NH. The field disappears entirely for iron with about 3 wt. pct. silicon.

Fatigue mechanism map for single crystals of iron containing 0.5 wt% silicon.



2.2 Deformation of Polycrystals

Materials with High and Intermediate Stacking Fault Energy

The microstructural evolution during cold deformation of polycrystals has been analyzed according to a continuous subdivision of grains combined with a trapping of dislocations into low energy configurations. Good agreement has been found between the theoretical principles and observations for a number of medium or high stacking fault energy materials deformed in different modes. The metals investigated have been aluminium, copper, nickel and nickel-30% cobalt. The deformation modes were mainly rolling and torsion. A comprehensive review of this work has been given in three papers presented at a conference on microstructure and mechanical processing arranged by the Institute of Metals and held in Cambridge in March. Most of this work was limited to small and medium strains and further studies have concentrated on the microstructural evolution at higher strains.

Aluminium and copper have been studied after cold-rolling in the strain range 0.3-2.3 and it has been found that most of the microstructural evolution relates to deformation by stable polyslip. In copper, supplementary deformation takes place by concentrated shear and results in the formation of second generation microbands and shear bands. The structural evolution which is caused by stable polyslip consists of a continuous subdivision of the structure into volume elements delineated by dense dislocation walls or first generation microbands. Both at low, medium and large strains the dislocation walls and the microbands are related to the macroscopic sample axis. At low and medium strains the preferred orientation is 35-60° between the trace of the dislocation walls and the rolling direction in the longitudinal plane. At high strains most dislocation walls are oriented almost parallel to the rolling plane. Thereby a sheet structure is formed consisting of aligned subgrain walls. The spacing between such aligned walls is rather uniform and the volumes between the walls show alternating misorientations. These observations are in good agreement with the principle that the dislocations tend to arrange into configurations such as to decrease their energy per unit length of dislocation line. The microstructural evolution as observed in aluminium and in copper are shown in the figures below. In parallel to the microstructural studies, the texture evolution as a function of strain is analyzed in order to correlate microstructural and textural observations. This may lead to an understanding of why the textural evolution is not identical in aluminium and copper when deformed by cold-rolling. A combination of microstructural and textural information is also used in a study of flow stress anisotropy of cold-rolled aluminium sheets.

The traditional view of the microstructure of deformed polycrystals has been that the individual grains have a rather homogeneous structure. This view was, to a large extent influenced by the predominant Taylor model for the plastic deformation of polycrystals. It is by now clear that the microstructure of real polycrystals is rather heterogeneous, which has very interesting implications for the theoretical description of polycrystal deformation. The Materials Department is actively engaged in updating the description of the microstructure of deformed polycrystals (this work is carried out in collaboration with the Danish Academy of Engineering, The University of Virginia and Sandia National Laboratories, California).

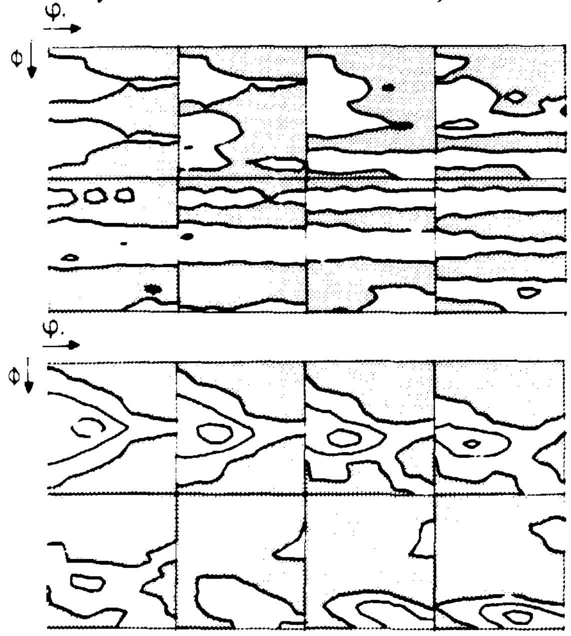
Transmission electron micrograph of the laminar sub-grain microstructure in 90% cold-rolled aluminium.

Brass

Materials with low stacking fault energy (SFE) like brass have a textural and microstructural evolution which is different from that in materials with high SFE. The intimate relation between texture and microstructure has been demonstrated in rolled brass plate which was bent during rolling and then straightened. The total added strain from bending and straightening was ≈ 0.05, which is small compared to the rolling strain of 0.5. Nevertheless, the texture of the bent and straightened plate was completely different from that of material rolled to the same strain without bending and straightening - as shown in the ODF (orientation distribution function).

The explanation is found in the microstructures. The formation of the normal brass texture is conditioned by a microstructure with one system of »bundles«, consisting of a composite of twin and matrix lamellae, in about 50% of the grains. Bending and straightening destroys the microstructure (replacing it by a complex structure with two or three systems of twins) removing the microstructural conditions which lead to the normal brass texture. In this way an otherwise insignificant added strain produces a dramatic change in the textural evolution.

Difference between the ODF for brass rolled with bending followed by straightening (top) and brass rolled without bending (bottom). For each material, 8 sections of the ODF with 5° between each, are shown.



2.3 Modelling of Internal Stresses in Metal Matrix Composites

The average internal stresses generated in metal matrix composites (MMCs) by differential thermoelastic and plastic deformation of the constituent phases are estimated by computer calculations based on the Eshelby method of ellipsoidal inclusions. The calculations were carried out in collaboration with Department of Materials Science and Metallurgy, University of Cambridge, UK. The main objective is to develop improved estimates for high volume fractions f of inclusions, particularly short fibres represented as ellipsoids. The problem at high f values is that the physical picture of the conventional Eshelby models is structurally biased: it involves a connected matrix phase with unconnected inclusions. This bias becomes unrealistic at high f values, where the inclusions touch each other to the extent that they begin to form a connected phase.

The mean field variant of the usual Eshelby model was therefore given a further extension to remove the structural bias. This led to a »variable constraint model« (VCM), in which the inclusion-matrix constraint is represented by a homogeneous »comparison material« with adjustable stiffness tensor C_o. The VCM is ideally suited for investigating the problem of bounding the MMCs inelastic properties and internal stresses. It ignores the exact positions of the inclusions but provides a spectrum of possible estimates depending on the assumed C_o tensor. The most important estimates are the »upper bound« estimates ($C_o = C_I$), the »lower bound« estimates ($C_o = C_M$) and the »self-consistent« estimates ($C_o = C_c$). Here C_I, C_M and C_c are the stiffness tensors of inclusions, matrix and composite, respectively.

It turns out that for spherical particles and short fibres, represented as ellipsoids, the »upper bound« estimates cross the lower bound estimates of the average internal stresses and/or overall thermal expansion coefficients. Hence these estimates evidently cannot be rigorous bounds. This rather surprising result is not consistent with the fact that the VCM delivers rigorous bounds in the continuous-fibre case. By exploiting a recent analytical expression of Eshelby's S-tensor for transversely isotropic media, the VCM provides a convenient iterative scheme for calculating »self-consistent« estimates. At high fibre contents such estimates would seem to be more reliable than the conventional »lower bound« estimates or the »upper bound« estimates.

2.4 Metal Matrix Composites

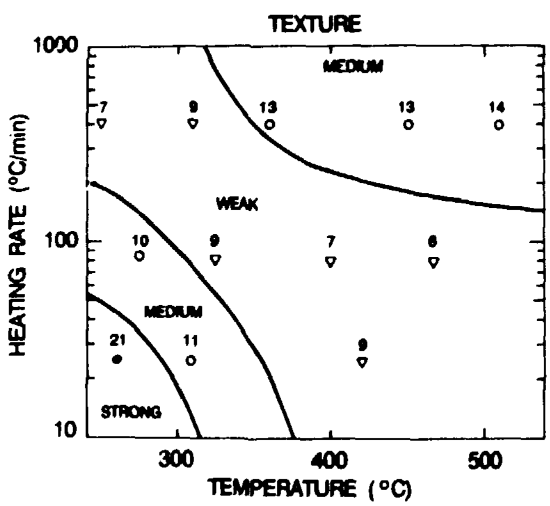
This programme concentrates on aluminium-based composites manufactured by powder metallurgy. The composites are reinforced with short fibres or particles of silicon-carbide (SiC). The objective of the work is to study the mechanical, physical and chemical properties of Al-SiC composites and model their behaviour. Composites are studied as-manufactured as well as during and after the application of loads. Part of the programme is described in the following subsections. In addition, transmission electron microscopy (TEM) studies have been carried out (in collaboration with the Department of Engineering, University of Cambridge, UK) to study the dislocation configurations after cooling and after straining in tension. This information together with a careful characterization of the microstructure, texture and internal stress state form the basis for mechanical modelling. An important technological achievement has been the development of sol-gel techniques for coating of SiC fibres or particles in the as delivered state. (A patent on this process is pending). Composites with coated fibres have been heated at temperatures far above the matrix melting point and significant thermal stability has been demonstrated in comparison with composites containing uncoated fibres. Also an improved distribution of the reinforcement during processing leads to improved mechanical properties. The composite development in combination with the basic studies involves the Department both in the industrial manufacturing of metal matrix composites and in their practical application.

Texture Control in Al/SiC

An important scientific as well as practical aim is to be able to model and control the evolution of texture and grain size during thermomechanical processing. For a given material, this requires knowledge of the effect of a number of process parameters such as strain, strain rate, temperature and heating rate. As the effects of these different process parameters are not independent, it is indeed not straightforward to predict the resulting textures and grain sizes. Experiments covering a very large parameter range are needed. For 90% cold rolled Al + 2 vol% SiC whiskers, the effects of variations in annealing temperature and heating rate (dT/dt) have been studied. This material was chosen because it belongs to the class of new matrix composites reinforced with short fibres or particles; these are emerging as potential engineering materials. Also earlier investigations have shown that the annealing temperature has a remarkably large effect on the recrystallization texture and grain size. As a result of the present investigation, maps relating the effects T and dT/dt on texture and grain size are constructed.

The annealing texture of the Al + 2 vol% SiC material is characterised by a strong {100} < 013 > orientation (in the following referred to as the N-orientation). Besides this component, the other components are fairly randomly distributed in the orientation space. The strength of the N component depends strongly on T and dT/dt. This is shown in the form of a map in the figure below. Also the recrystallized grain size varies with T and dT/dt. Maps of this type can, in combination with investigations of texture/microstructure relations, for example by the electron back scattering technique, be used to

This map shows the strength of a given texture component which results from annealing at different heating rates and to different temperatures; aluminium containing 2 vol% SiC whiskers. Such maps may be used as guidelines in deciding annealing treatments to be used in industrial processes in which specific textures are required.



get basic information about the nucleation and growth processes. More straightforwardly, the maps can also be used as guidelines to help choose annealing conditions to obtain specific textures and microstructures. If, for example, a material with a small grain size and a weak texture is wanted, a high annealing temperature (T > 400°C) and low heating rate (dT/dt < 200°C/min.) should be used. Or if a large grain size and a strong texture is the optimum, both T and dT/dt should be low (e.g. T < 300°C and dT/dt < 50°C/min.). For predictions outside the original parameter range the maps may also be used at least to give crude guidelines. In the present series of experiments, annealing using dT/dt = 3.3°C/min. and T = 400°C was also tried. According to the maps, a grain size just above 200 μm and a N-texture about 10 × random should be expected. Experimentally, grain size was determined to 242 μm and the texture to 11.2 × random.

Internal Stresses in MMC

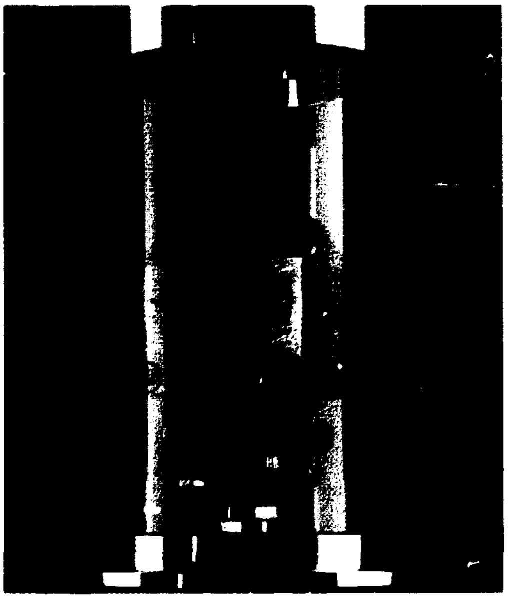
Most MMC materials possess internal strains due to the mismatch in the coefficient of thermal expansion between the different phases in the material. An important tool to study these stresses is the neutron diffraction technique by which the lattice strain of the different constituents can be studied.

Recent studies of internal strains in such MMC materials by neutron diffraction have concentrated on studies of room temperature ageing effects in the precipitation hardening system Al-4Cu reinforced with SiC particulates, and studies of internal stresses in Al/SiC whisker reinforced materials.

During 1990 a dedicated instrument for such studies of internal stresses has been completed and implemented at the DR-3, TAS-8 facility. Further, a new version of a stress-rig has been constructed, allowing both thermal and mechanical loading of composites during the neutron diffraction experiments. Test specimens can now be exposed to thermal loadings up to ≈ 400°C and mechanically up to a total uniaxial load of 10kN. This allows for studies of creep and relaxation phenomena at elevated temperature as well as low cycle fatigue of MMC's.

With the new instrument and stress-rig, coming experiments will focus on elevated temperature creep experiments on Al/SiC whisker composites. Further studies will include strengthening mechanisms and aging effects in Al-4Cu/SiC particulate composites and studies of internal stresses in Al-Mg/Al_2O_3 fibre composites.

An Al/SiC composite test specimen mounted in the sample chamber of the newly developed stress rig in which internal stresses are determined by neutron diffraction. In-situ testing, at temperatures up to 400°C, is carried out with the specimens surrounded by an inert atmosphere.



Transmission electron micrograph of a surface-coated SiC fibre in an extruded aluminium matrix. The magnification is approx. × 25,000.

Mechanical Behaviour of MMC

Knowledge of the effect of powder metallurgy (P/M) processing parameters on the microstructure of Al-SiC composites and the established correlation between structural parameters and mechanical properties in this material has allowed the P/M process to be optimized with respect to the mechanical properties.

Ultrasonic wet blending in organic solvents is effective in de-agglomerating and dispersing SiC whiskers within an Al matrix while reducing the whisker breakage. This treatment increases E modulus, $\sigma_{0.2}$ and σ_{uts} in an extruded Al-10 vol% SiC_w by 4%, 23% and 16% due to a more homogeneous whisker distribution, better whisker alignment and higher aspect ratio.

The alumina sol gel technique using inorganic colloidal sol produces a coating layer on the SiC whiskers (particulates). A uniform whisker distribution, better whisker alignment and an increased concentration of alumina particles is achieved. As a result, an increase of 14%, 20% and 29% in E, $\sigma_{0.2}$ and σ_{uts} without loss of ductility for an Al-10 vol% SiC_w has been obtained. Also, the thermal reaction between SiC_w and molten Al matrix, which degradates SiC_w, is reduced due to low diffusivity of Al and Si through complex layers of alumina at the interface.

A combination of a sol gel treatment and wet blending provides a further possibility of improving microstructure and properties. In Al-10 vol% SiC_w up to about 50% increase in $\sigma_{0.2}$ has been achieved.

Work is in progress to evaluate the effect of the coating layer on the structural characteristics during deformation and mechanical properties of the composites.

2.5 Deformation of Polymer Matrix Composites

Reinforcement of polymers with strong and stiff fibres is a very efficient method for improvement of the mechanical performance of polymers. The properties of these polymer matrix composites under mechanical, thermal and chemical (corrosive) loadings are studied by several techniques. The aim is to establish knowledge of the behaviour of polymeric composites as a basis for the use of the materials in design. The special direction of the work is towards long term performance under demanding conditions.

Creep Behaviour

The studies involve creep properties of polymeric composites of glass fibre/polyester and carbon fibre/epoxy. Both experiments and modelling are carried out with the microstructural parameters being volume fraction of fibres, fibre length/diameter ratio and fibre orientation. The models have been established and are valid for all fibrous composiste materials. The models account for fibre volume fraction, length/diameter ratio and will be further developed to include fibre orientation.

Experiments on pure epoxy are performed at 60°C and 90°C and at various stresses; the creep curves of strain versus time show visco-elastic behaviour with a decreasing creep rate. The analysis and description of the curves are made by an empirical equation, which relates to a physical model of elastic elements and (fully) plastic elements; the relevant descriptive parameters are determined by a fitting procedure. The aim is both to generate engineering descriptions of creep curves, and to extract the deformation mechanisms.

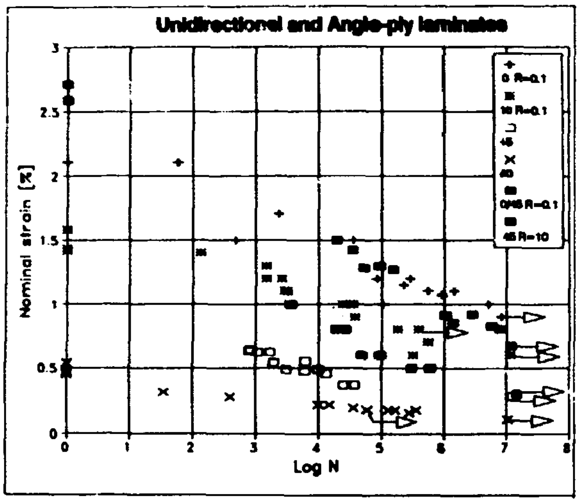
Experiments on carbon fibre reinforced epoxy are performed at 90°C. The composite materials are pure epoxy, epoxy with fibres oriented 90° to the loading axis, and epoxy with fibres oriented 45° to the loading axis. From the creep curves an estimate is made of the creep rate and this is plotted versus the creep stress. There is an approximate straight line relationship between the logarithm of the creep rate and the stress. The 90°-composite is stronger than the pure epoxy by a factor of about 1.2, and the 45°-composite is stronger by a factor of about 2.1.

Fatigue Behaviour

The studies involve a detailed investigation of glass/polyester laminates with various fibre orientations: 0°, ±10°, ±45°, ±60°, and 0°/±45°. This family of materials is relevant to practical wing blades for wind turbines.

Experiments are made at room temperature and at a frequency of 5 Hz. the loading is both tension-tension (R = 0.1) and compression-compression (R = 10). The fatigue strength decreases with increasing fibre orientation angle, with the 0°/±45°-composite being very close to the 0°-composite. The loading conditions (R-ratio) show that the 10°-composite is stronger in tension than in compression while the opposite holds for the 45°-composite.

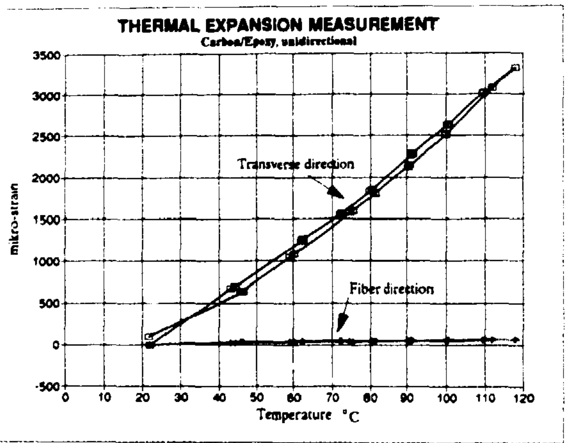
Measurements of stiffness (E-modulus) show that significant reduction occurs, especially towards the end of the lifetime. This is being used to define fatigue curves for a given stiffness reduction, which can form the basis to establish design limits. This has special interest for wing blades for wind turbines.



Fatigue data, in both tension and compression, for glass/polyester laminates.

Strain Limits for Laminates

The difference between the manufacturing and operating temperatures for a polymer composite component (thermoset or thermoplastic), results in residual stresses due to the difference in thermal expansion between fibres and matrix and to the anisotropy of the thermal expansion coefficient for the composite material. The influence of these residual stresses on the static strength properties are studied for both carbon/epoxy and carbon/PEEK laminates in the temperature range -50 to 150°C. The temperature-dependent thermal expansion coefficients are established for the whole temperature range, and tensile tests are performed with simultaneous observation of crack formation on the specimen edges. The thermal stresses in the specimen are modelled by finite element techniques and used in the evaluation of the test results.



Measured thermal expansion data for a unidirectional carbon/epoxy composite. Large residual stresses in single ply laminates are caused by differences in thermal expansion along the fibre direction compared to the transverse direction.

2.6 Irradiation Defects - Fusion Materials

Irradiation of metals by energetic particles removes atoms from their lattice sites, leaving vacancies and creating interstitial atoms. Very energetic particles can, in addition, induce nuclear reactions and generate gaseous and non-gaseous impurity atoms, changing the composition of the irradiated metal or alloy. The created defects may diffuse together to produce nanometer-size defect clusters; their presence changes mechanical, thermal, magnetic and electrical properties. The fundamental study of such defect clusters and of the generated microstructural changes lies at the heart of physical metallurgy; the diffusion process and their consequences are closely related to temperature effects and property changes not involving irradiation. The work in this area comprises both theoretical and experimental efforts, the applied experimental techniques being transmission electron microscopy, positron annihilation, and small angle neutron scattering.

These techniques are being developed to give fundamental information close to the atomic level. This information is necessary to the other areas of our work in which mechanical properties of materials for future fusion reactors are being studied.

Displacement Damage and Helium Effects in Copper and Copper Alloys

It is a matter of serious concern that both physical and mechanical properties of materials exposed to an intense flux of fusion (14 MeV) neutrons may be significantly degraded because of the production and accumulation of displacement damage and helium during irradiation. The prime objective of the present programme is to study these effects in copper (OFHC grade) and copper alloys (Cu-Al_2O_3, Cu-Cr-Zr and Cu-Ni-Be) which are being considered as candidate materials for divertor components in fusion devices such as NET and ITER. Recently an additional alloy (Cu-ZrO_2) has been included in the programme. Both tensile and fatigue specimens of copper and copper alloys have been irradiated with fast neutrons in DR-3 (Risø). So far, only tensile specimens have been irradiated with 600 MeV protons (at the Paul Scherrer Institute, Switzerland). Pre- and post-irradiation mechanical properties (tensile and low cycle fatigue), electrical conductivity and microstructures of these materials will be determined.

In order to examine the effects of recoil spectrum on void swelling, microstructures of copper specimens irradiated with fast neutrons and spallation neutrons have been studied using transmission electron microscopy (TEM). Because of the limited number of experiments, no firm conclusion can be drawn at this stage. However, the indication is that the swelling rate in the case of spallation neutron irradiation may be somewhat lower than that caused by fast neutron irradiation.

To study the effect of helium generation rate on cavity formation and transport of helium to grain boundaries, copper specimens have been implanted at different rates in an accelerator in Karlsruhe, FRG. The cavity and dislocation microstructures of these specimens have been studied using TEM and positron annihilation technique. The results are being evaluated.

A new rig with accurate temperature measurement and control facilities has been designed and is being fabricated for irradiation at temperatures in the range of 200-450°C.

Difficulties in the manufacturing of the new Cu-ZrO_2 alloy with an acceptable microstructure made it necessary to characterize the microstructure of the alloy at various stages of the fabrication procedure.

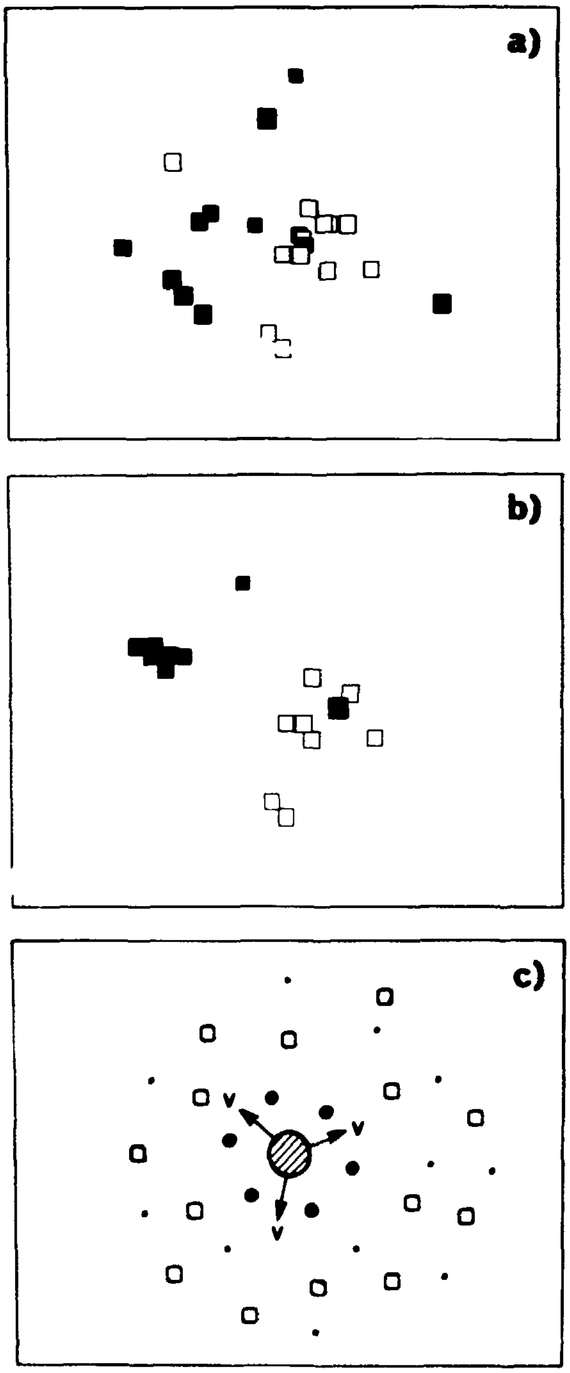
Observation of Defect Structures in Copper, Gold and Cu_3Au

600 MeV proton irradiations produce primary recoil energies of several MeV. The resulting cascade and sub-cascade microstructures have been investigated by high resolution weak beam electron microscopy. The visible defect clusters are typically 0.5 to 5 nm in diameter. It was therefore necessary (in collaboration with Paul Scherrer Institute, Switzerland) to reassess the accuracy of the available electron microscopy techniques. The size and visibility of the images of these small clusters varies with image technique and operating conditions These variations are now well documented and used to determine »absolute« defect sizes and concentrations.

Production Bias and Temperature Dependence of Void Swelling

Recently it was proposed (in collaboration with Atomic Energy of Canada Ltd., Pinawa, Canada) that instantaneous clustering of vacancies and interstitials produced in a displacement cascade yields a production bias which may play a dominant role in controlling void swelling. As a first application of the model, the temperature dependence of the steady-state swelling rate has been calculated.

Clustering of interstitials and vacancies from a displacement cascade; »production bias«.



Two sharply separated temperature regimes occur with low swelling rate at lower temperatures and high swelling rate at the peak swelling temperature. The steady state swelling rate at the peak temperature can be a factor of ten higher than that expected at lower temperatures. The high swelling rate regime is dominated by the production bias whereas the dislocation bias determines the low swelling rate regime. The transition from the low to the high temperature regime is governed by the activation energy nearly equal to that for self-diffusion.

The production bias provides an explanation for the high steady-state swelling rate, despite the very small fraction of free point defects surviving under cascade damage conditions. One does not have to rely on the arbitrary assumption of a very large dislocation bias which must be very different under cascade and non-cascade conditions.

Morphology of Collision Cascades

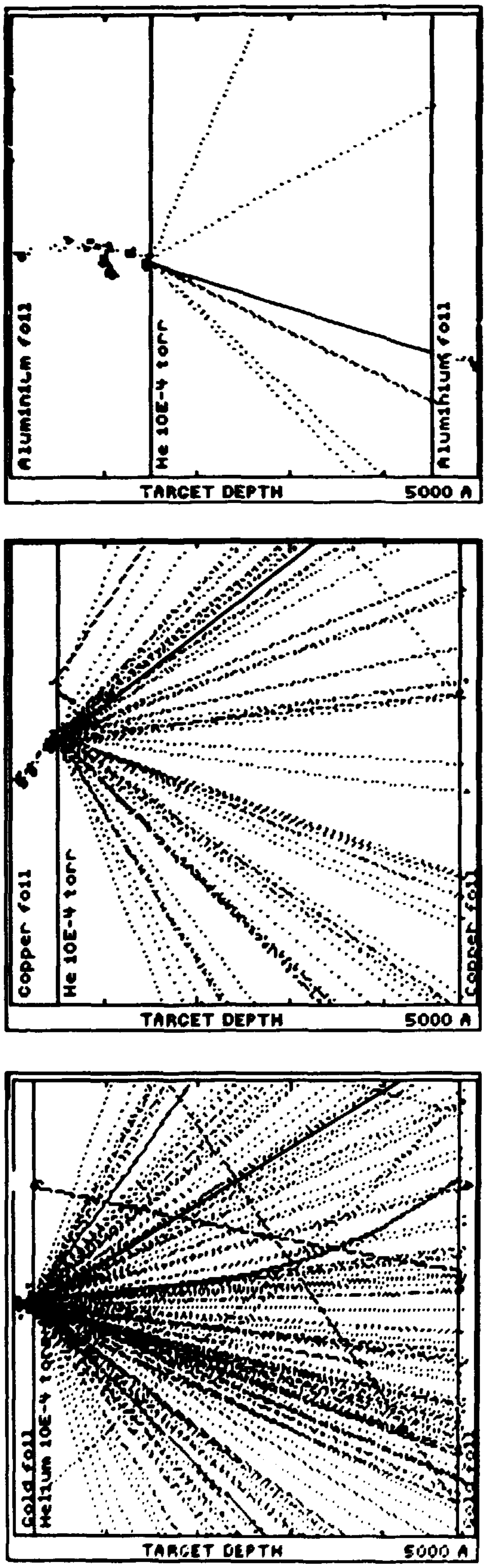
The interaction of high energy particles with a crystalline solid produces collision cascades containing displaced atoms and vacant lattice sites. The morphology of the cascade region in the material depends on the energy of the primary recoil atom that initiates the cascade of collisions. At high recoil energies, multiple, widely-separated damage regions, commonly referred to as subcascades, can be created in a single recoil event. The morphology and spatial distribution of the cascades and subcascades are likely to affect microstructural evolution and damage retention during irradiation.

Towards a better understanding the influence of cascade and subcascade production on microstructural evolution, efforts have been made to determine the threshold energy for subcascade formation, the number of subcascades per recoil per unit energy and the average spacing of subcascades in fcc and bcc metals. In collaboration with Pacific Northwest Laboratory, Richland, USA, computer simulated collision cascades and subcascades were generated with the binary collision code MARLOWE.

Compared on the basis of reduced damage energy, metals of the same crystal structure have a subcascade threshold at the same reduced energy. The number of subcascades per unit reduced damage energy is about the same for metals of the same crystal structure, and the average spacing of subcascades is about the same in units of lattice parameters. Comparisons between subcascade threshold energies and average recoil energies in fission and fusion neutron environments show the spectral sensitivity of the formation of subcascades.

Monte Carlo Simulations of Cascades in Thin Foil Irradiations

The computer code, TRIM, has been used to simulate cascade damage due to 20 keV, 200 keV and 2 MeV self-ions in isolated thin foils, stacked thin foils, and bulk specimens of Al, Cu and Au. Changes in foil thickness have been shown to cause large changes in energy loss processes. For stacked thin foils, separated by a vacuum or helium gap, the production of cascades has been shown to be rather different to that occurring in bulk. A major difference is that secondary recoils may be emitted from one thin foil across the gap into the second foil. The secondary recoils are emitted in divergent trajectories. As a result, many sub-cascades produced in thin foil irradiations do not occur in sub-cascade groups. The results of the simulations have an important bearing on experimental investigations.



Computer simulation of self-ion recoil damage typical of 14 MeV fusion neutron irradiation in thin foils of aluminium, copper and gold.

2.7 Solid Electrolytes - New Materials and Methods

Solid electrolytes have many uses as ionically conducting membranes in fuel cells, batteries, gas- and ion-sensors, electrochemical gas pumps, electrolysers and thermoelectric generators.

The ongoing research in ionically conducting solids continues a well established research theme of the Department. It is also constituting the more fundamental background for the development of Solid Oxide Fuel Cells (see Section 4.3 for a detailed description of SOFC).

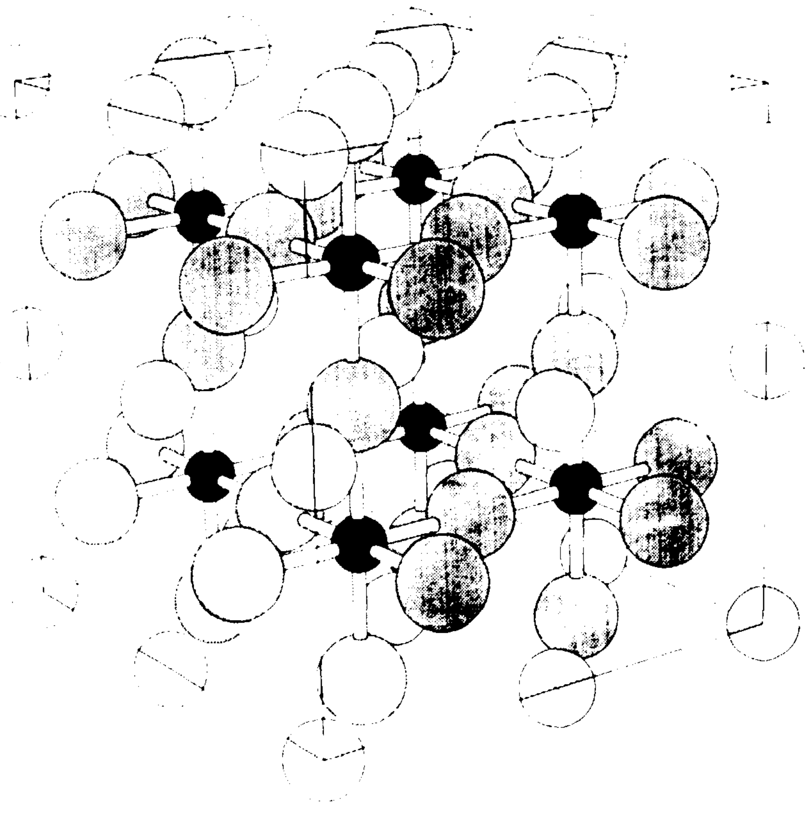
Thin ceramic coatings on metal-, alumina-, and zirconia-substrates have been made by spray pyrolysis, and characterised by SEM and X-ray powder diffraction.

The formation by solid state reactions of lanthanum- and strontium zirconates and the solid electrolyte properties of such likely impurity phases in SOFC's have been investigated. These intermediate phases form quite easily, and have ionic conductivities 1 to 3 orders of magnitude lower than »state of the art« SOFC materials.

As an alternative to conventional conductivity measurements a four-probe high temperature van der Pauw method has been developed. The applicability of this method has been verified by comparing conductivities of approx. 200 micron thick yttrium-stabilised zirconia ceramic measured in 7 different geometries.

A fully automated data collection and data treatment procedure for alternating current conductivity determination (25-1100°C) has been built. A »round-robin« series of tests on ionic conductivity in fluorite type oxides is being carried out under the auspices of the International Union for Pure and Applied Chemistry; 12 other laboratories around the world are participating in the tests.

Model of the perovskite crystal structure. SOFC-electrodes and interconnects are typically of this type.



2.8 Polymer Chemistry and Physics

An increased and detailed understanding of the relationship between the molecular- and microstructure of polymers and their properties is important for basic and applied research as well as for technological development. The scientific goal for this research is to understand these relationships in such a way that properties of pure polymers, polymer mixtures or blends as well as polymer solutions can be predicted. In order to achieve this goal, research activities within the areas of polymer synthesis, polymer analysis and characterization, and modification of polymers are necessary. It is believed that this combination of research activities facilitates the production of tailor-made polymer materials with desired properties.

PEPS: A New Solid Matrix for Peptide Synthesis

The principle of anchoring molecules onto a solid matrix, which helps to »keep track« of the intermediate products during chemical transformations, is known as *Solid-Phase Synthesis* or the *Merrifield Technique*. Established methods for the solid-phase assembly of amino acids into peptides (protein-like molecules) employ a beaded matrix of crosslinked polystyrene. Based on the recognition that most operations are identical in the synthetic cycles of solid-phase peptide synthesis, a new matrix, PPS, was recently introduced to facilitate and speed up the preparation of large numbers of peptides. This work was carried out in collaboration with Merrifield Laboratory, The Rockefeller University, U.S.A. This matrix is comprised of a polyethylene, PE, film with pendant long-chain polystyrene, PS, grafts. The loading capacity of such a film is equally high to that of a beaded matrix but PEPS has the additional flexibility to suit multiple syntheses simultaneously. Thus, in a new configuration for solid-phase peptide synthesis, the PEPS film is fashioned in the form of discrete, labelled sheets, each serving as an individual compartment. During all the identical steps of the synthetic cycles the sheets are kept together in a single reaction chamber permitting the concurrent preparation of a multitude of peptides at a speed close to that of a single peptide by conventional methods. The development of PEPS is based on the invention of a new γ-radiation-induced grafting procedure (see Section 3.9) and patent protection of the whole concept has been applied for.

A polyethylene-polystyrene (PEPS) stick used in the synthesis of peptides. The reaction is followed by monitoring the intensity of Ruhemann's purple formed in the Kaiser ninhydrin reaction.

Comb-Shaped Polyesters

Comb-shaped polymers have evenly spaced, equally long alkyl side-groups attached to the linear main-chain backbone. Comb-shaped polyesters in which the backbone is a polyester chain are interesting structural models for graft copolymers or biomembranes, and have potential applications as membrane materials or as carriers for light sensitive groups useful for optical recording and data storage. Such polyesters offer extended possibilities in structural design through controlled variation of building blocks and are thus well suited for fundamental studies of relationships between structure and properties, e.g. thermal and mechanical properties.

In collaboration with CNR, Dept. of Chemical Engineering, University of Pisa, Italy, a comprehensive and basic synthesis work was undertaken aiming initially at procedures resulting in high molar mass polyesters. A successful preparation and detailed characterization - solid state as well as solution behaviour - of an extended homologous series of very pure aliphatic diphenyl ester precursors was accomplished and thoroughly discussed. A new polycondensation procedure resulting in a range of comb-shaped polyesters with qualitatively good film- and fiber forming properties has been developed. The new polyesters were analyzed by intrinsic viscosity and size exclusion chromatography with both refractive index and laser light scattering detection. Absolute molar masses and average degrees of polymerization (DP) were established. The achieved DPs, generally around or above 100, are in line with commercial (*not comb shaped*) polyesters and considered very acceptable. Another solution characteristic, ^{13}C nuclear magnetic resonance (NMR) spectroscopy, has confirmed the anticipated structural features.

All the comb-shaped polyesters are crystalline as shown by X-ray diffractometry, infrared spectroscopy and differential scanning calorimetry. In case of the aromatic polyesters, the crystallinity is exclusively due to the octadecyl side-group which crystallizes in the hexagonal form. The aliphatic comb-shaped polyesters, on the other hand, have a much more complicated behaviour. Here the main-chain takes part in the crystallization as well. Detailed investigations of the structural influence on crystallinity, thermal and mechanical behaviour of the aliphatic polyesters are in progress.

Phase Behaviour of Block Copolymers

The temperature at which a phase transition, e.g. the melt transition in a substance takes place, is in general determined by the balance between entropic and energetic factors. At high temperatures entropic factors dominate and disordered states like melts are favoured. At lower temperatures energetic factors dominate and ordered states like crystalline solids are favoured.

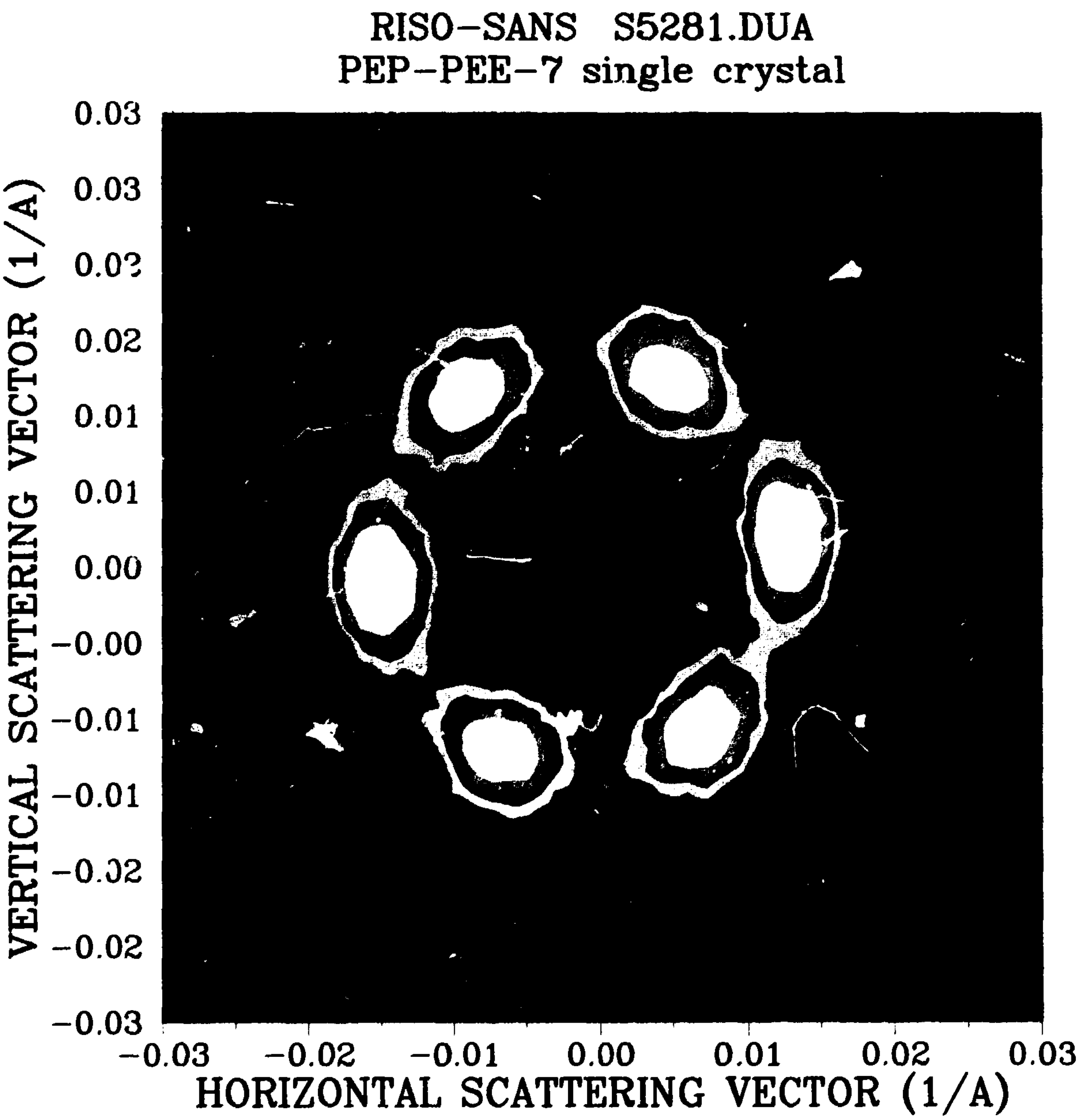
A similar balance between entropic and energetic factors determines whether a two-component system is mono- or biphasic. Entropy of mixing is an important driving force for the mixing of many low molar mass substances. In polymers the entropy of mixing effects are suppressed due to the size of the molecules and two different polymers are in general not miscible unless the molar mass is low.

A block copolymer consists of two different kinds of polymers linked together by a chemical bond. Macroscopic phase separation is thus not possible. Instead so-called microphase separation occurs. Block copolymers typically contain domains rich in one or the other of the components. The size of the domains is dictated by the molecular architecture and is on the order of 10 nm.

The balance between entropic and energetic factors is particularly delicate in certain *soft* materials such as microemulsions, micellar solutions, and block copolymers. Lacking the short range given by the atoms in conventional crystalline solids, these mesomorphic substances can fill space through the formation of intriguing microstructures. Block copolymers are particularly well suited for studying these phenomena because the molar mass and composition of a sample can be precisely controlled in the chemical synthesis. This allows access to different regions in the phase diagram.

The particular model diblock copolymer we are studying in collaboration with the Department of Chemical Engineering and Materials Science, University of Minnesota and The Department of Solid State Physics, Risø consists of polyethylene-propylene, PEP, and polyethylethylene, PEE. The PEP-PEE diblock copolymer phase diagram is mapped by dynamic shear modulus and small angle neutron scattering (SANS) measurements. A sample with molar mass 10^5 g/mol containing 77 vol% PEP gives rise to a microphase separated ordered material where the ordered state morphology is hexagonally packed rods which is in accordance with current theory. A fascinating aspect of the PEP-PPE materials is that long range order can be induced by application of a shear field. Centimetre size single crystals can be produced and the resulting anisotropy studied by SANS. In a sample with molar mass slightly lower than 10^5 g/mol containing 65 vol% PEP we have observed three different microphase separated ordered states simply by changing temperature. The disordered states is accessed in this material at a temperature of 175°C.

SANS data from a PEP-PEE single crystal containing 77 vol% PEP. The centimetre-sized crystal was produced by the application of a large amplitude shear field during cooling from the disordered state.



2.9 Characterization of Microstructure

Electron Microscopy

The relation between structure and properties plays an important role in many of the Department's projects. Two transmission electron microscopes - JEOL 2000FX and JEOL 100C - and one scanning electron microscope - JSM 840 - are available for structural analysis. All microscopes are equipped with energy-dispersive X-ray spectrometers for chemical analysis; the JEOL 2000FX furthermore is equipped with an electron energy loss spectrometer.

Microstructural work has been performed for a number of the Department's own projects. This work is reported under the individual projects. During the year, microstructural analysis has also been done for a number of industrial customers.

In collaboration with the Department of Solid State Physics, Risø, the structure of sputterdeposited layers of the high temperature superconductor $YBa_2Cu_3O_{7-x}$ on MgO (100) single crystal substrates have been studied by TEM. An example of a less satisfactory thin film with subgrains, some of which are rotated 45° with respect to the majority of subgrains, is shown in the figure below.

Sputter-deposited layer of the high temperature superconductor $YBa_2Cu_3O_{7-x}$ onto a single crystal of MgO. Transmission electron micrograph, magnification × 40,000.

Solid inclusions of sodium in an aluminium matrix. In the series of micrographs, voids at the sodium/aluminium interface were seen to have moved, coalesced and facetted during observation in the electron microscope.

In collaboration with Ecole Polytechnique Federal de Lausanne (Switzerland), solid inclusions of sodium in aluminium have been studied by a variety of electron microscopy techniques. Shrinkage voids occurred within the sodium particles. These voids were seen to move, coalescence and facet during observation in the transmission electron microscope. The in-situ observations provided valuable information on surface energies, void/bubble migration and coalescence mechanisms in immiscible systems.

A Tunnelscope 2400 scanning tunneling microscope has kindly been lent to the Department for some months by the Danish producer, Struers A/S. The Department has thus had an opportunity to explore the possible applications of a tunneling microscope within our field of work. An example of a study of slip lines on the surface of rolled copper is shown in the figure to the right.

Electron Back Scattering Patterns

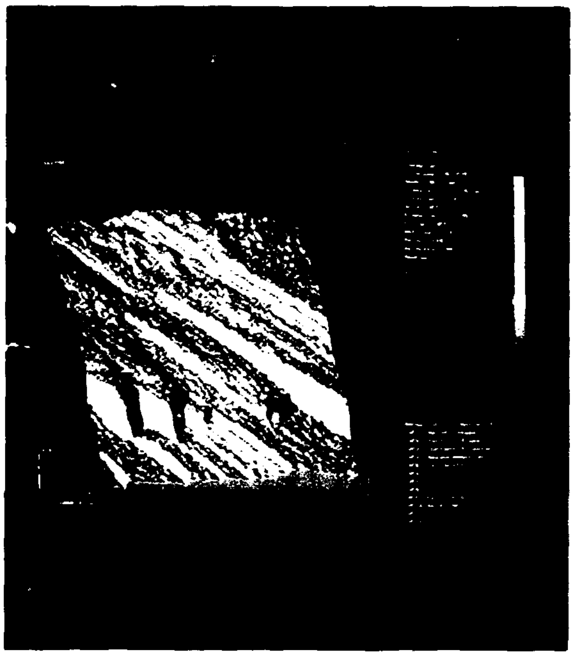
A very powerful technique for measurements of crystallographic orientations in selected local areas of the microstructure is the electron back scattering pattern, EBSP, technique. Advantages with this technique are that it has a very good spatial resolution ($\approx$ 0.5 μm in diameter) and that it can cover large sample areas ($> \approx$ 1 cm^2). EBSP equipment is attached to the JSM-840 scanning electron microscope, and a computer program for analysis of the patterns has been developed. Compared with the commercially available computer program, advantages with the Risø program are that it can be applied for any crystal symmetry, that it requires no knowledge of electron diffraction maps, that it can be used for EBSPs with relatively low contrast and that the indexing is very precise.

A preliminary investigation has shown that it is possible to use image processing procedures for computer identification of bands in an EBSP. Fully automatic measurement and indexing of EBSPs may be possible and is presently being developed. During the year, the EBSP technique has mainly been used for measurements of grain size/orientation relationships in recrystallized Al-2 vol% SiC and investigations of nucleation and growth in commercially pure Al.

Neutron Diffraction and Scattering

Three neutron techniques have been used for characterization; internal strain, texture and small angle neutron scattering.

The internal strain instrument has been upgraded to allow measurements with very high spatial resolution. This is done by incorporation of 4 beam definers, 2 between the monochromator and sample and 2 between sample and detector. The beam definers are constructed as adjustable aperatures with sideways translations. The spatial resolution thereby achieved is 10 mm^3 for a bulk cylinder and 1 mm^3 for a thin plate of a typical



Study of slip lines on the surface of rolled copper; scanning tunneling microscope image.

solid oxide electrolyte material (yttria-stabilized zirconia, YSZ). With the new equipment, internal strain measurements have been carried out on a solid oxide fuel cell. Fuel cells are sintered at high temperature (1350°C) and residual strains build up during cooldown of the cells due to differences in the thermal expansion coefficients of the cell materials. The measured cell was a 42 by 42 mm plate with three 0.1 mm thick layers: the anode (NiO mixed with YSZ), the electrolyte (YSZ) and the cathode (strontium-doped lanthanum manganite, LSM). The internal strain was determined for NiO, YSZ and LSM. It was found that NiO and LSM are in tension whereas YSZ is in compression, this is in agreement with plate theory calculations.

The internal strain equipment, in its more standard form, has mainly been used for determination of internal strain profiles in a large welded tube assembly, to investigate the effect of texture on the measured internal strain and to study ageing and other thermally induced effects in Al-Cu. Efforts have been concentrated on the design of a new stress-rig. With the new rig it will be possible to measure internal strains during tensile or compressive loading also at elevated temperatures.

The texture work has mainly been concentrated on investigations of a) recrystallization and texture optimization in Al + 2 vol% SiC, b) relaxation effects on texture development in Al + 2 vol% SiC, c) recrystallization kinetics in copper - a part of these results will be compared with results of stored energy measurements, d) texture/flow stress anisotropy relations in cross rolled Al and e) effect of large and small particles on the deformation/recrystallization texture development in Al.

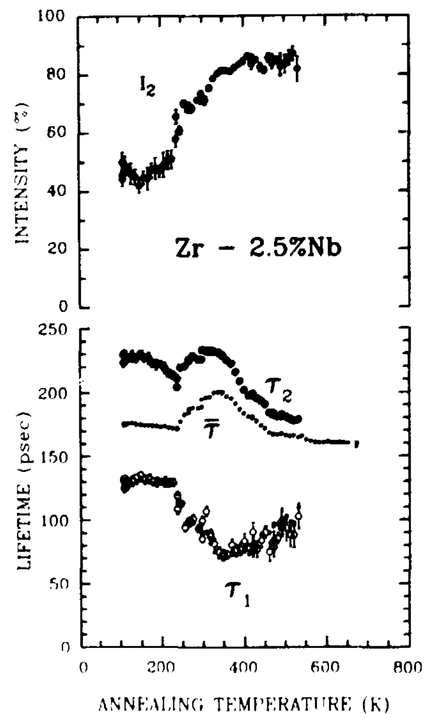
The small angle neutron scattering (SANS) facility has been used for studies of krypton filled cavities (so-called bubbles) in a nickel matrix. The aim is to determine the bubble concentration and size distribution as the bubbles grow during annealing. Complementary electron microscopy and positron annihilation measurements are carried out along with the SANS investigations. Finally SANS was used to study the phase diagram block copolymers. Polyethylene-propylene-block-polyethylethylene, of various compositions and molar mass were investigated especially for order-disorder and order-order transitions and ordered state morphologies.

Positron Annihilation

In studies of defect properties of metals it is useful to apply different experimental techniques which may provide complementary information. One of the defect sensitive techniques is the so-called positron annihilation technique (PAT) in which the sample under investigation is bombarded with positrons (antiparticles to electrons). Their annihilation with electrons in the sample results in the emission of gamma quanta. The time and angular correlation of these gammas can be measured and gives experimental information about electron density and momentum in the sample in the vicinity of the positrons. Since positrons are attracted to vacancy type defects (e.g. single vacancies, vacancy clusters, vacancy-impurity clusters etc.) the presence and characteristics of such defects can be detected by PAT, because at one such type of defect the electron density and momentum normally are different from those of the bulk metal and of other defects.

A detailed series of positron lifetime measurements have been carried out on samples of zirconium 2.5wt% niobium that had been electron irradiated at low temperature (76K). In this collaboration with Chalk River Laboratories, Canada, a main objective was to seek information on the migration of vacancies which seems to take place at unusually low temperatures. It is suspected that the migration is facilitated by the formation of complexes between vacancies and iron impurity atoms. Isochronal annealing of the irradiated specimens was carried out and positron lifetime measurements made after each annealing step at 100K as well as in some cases at higher temperatures. Such sets of measurements make it possible to identify different types of defects that trap positrons strongly or weakly. The main results are in annealing stage I a decrease of the trapped-positron lifetime from 230 psec (close to that expected for a monovacancy) to about 210 psec at a sharp minimum at 240K, followed in stage III by an increase again to about 230 psec simultaneously with a strong increase of the positron trapping rateinto the defects. Above 350-400K both lifetime and trapping rate decrease. These and other positron data suggest that during irradiation and in stage I, vacancies become

Positron lifetime measurements from the isochronal annealing of electron irradiated zirconium containing 2.5% niobium.



decorated with impurities, while in stage III the strong increase in trapping rate probably is due to liberation of vacancies from, or loser binding to, other impurity complexes. Further experiments on high purity zirconium are planned.

In positron lifetime measurements an important problem is the accurate determination of the contributions to the measured spectra, arising from positrons annihilating outside the sample under investigation, mainly in the positron source material.

Before further analysis, measured spectra must be corrected for these contributions which depend on the source preparation technique as well as on the sample material surrounding the source. Measurements on a series of high purity, defect free metals have been carried out in collaboration with the Center for the Development of Nuclear Techniques, Algeria, and a general relationship for the correction has been obtained.

The lifetime of positrons depends on the electronic density experienced by the positrons. If trapped in a cavity containing gas (so-called bubbles), a positron's lifetime will therefore depend on the gas density. Taking advantage of this sensitivity it is possible to extract bubble sizes and concentrations as well as the gas density in the bubbles and in this way, non-destructively, to follow the annealing behaviour of the bubble population in a specimen after irradiation or gas implantation. Such studies are carried out in parallel with electron microscopy and small angle neutron scattering (see section on neutron scattering). Measurements have been made in collaboration with KfK Karlsruhe, FRG (helium in copper) and KfA Jülich, FRG (helium in nickel, iron and steel) and with the Solid State Physics Department, Risø (krypton in nickel).

Characterization of Macromolecules

The behaviour of macromolecular systems is strongly dependent on the chemical structure and the length of the polymeric chains (molecular weight). Furthermore, significant modifications are possible by addition of small amounts of low molecular weight component. Characterization methods must therefore include chemical as well as physical methods.

Fourier-transform infrared, FTIR, spectroscopy provides information about the chemical structure. FTIR is a vibrational spectroscopy and is often referred to as a »fingerprint« method. In several cases, however, a FTIR analysis unequivocally identifies the investigated sample. The recently installed FTIR spectrometer has many attractive features. The coupling to a microscope allows identification of single (polymer) fibers or makes studies of very small and selected sample areas possible. Different sample holders are especially suited for surface studies. Surfaces of planar and soft, flexible samples, e.g. polymer films, can be investigated by attenuated total reflectance, ATR, and surfaces of rough, hard or powdered materials can be studied by diffuse reflectance infrared Fourier-transform spectroscopy, DRIFTS. A special detector making use of the photoacoustic, PA, emission generated when enclosed samples are irradiated with infra-red light has proved very promising for the analysis of polymer based composite materials especially when they contain carbon fibers. In general, heavily carbon loaded samples are very difficult or impossible to analyze by FTIR because the incident infra red light is absorbed by the carbon. The high-performance polymers, polyetheretherketone, PEEK, or polyetherimide, PEI, with up to 60 vol% carbon fibers are easily and conclusively identified with PA-FTIR.

Information about relative molecular weight and molecular weight distribution are most easily obtained by size exclusion chromatography, a technique that separates molecules according to their size in solution. The conversion of relative molecular weights to absolute molecular weights is not simple, but a new technique combining a laser light scattering detector with a column giving total exclusion of the dissolved polymer, facilitates the determination of absolute molecular weights. This technique has been used for the characterization of block copolymers, random copolymers and homopolymers in organic or aqueous solutions.

The dynamic mechanical properties of macromolecular systems reflect the intrinsic properties of chain molecules and their interaction with surrounding molecules. Temperature and frequency are important variables, and their effect on the mechanical properties has been used to study the effect of crosslinking, adhesion, cohesion and tack in model adhesives, curing kinetics of powder coatings, and formation of temporary gels in aqueous solution containing a hydrophilic/hydrophobic block copolymer.

3 Materials Engineering – Design and Testing

A thorough knowledge of the mechanical properties of engineering materials is essential for the design of advanced components and structures. Of special interest are materials for the oil and gas sectors and composite materials for wind mills, helicopters and lightweight pressure vessels as well as engineering ceramics. The research activities in Materials Engineering are centred around structural mechanics analysis of destructive and non-destructive materials testing procedures. A considerable number of the projects are carried out in close collaboration with Danish and European industrial partners.

Current research activities are concentrated on the following areas:

- Improvements of component design and service life predictions based on static, dynamic and fatigue testing results. Materials studied are steels, rapidly solidified Al-alloys, metal matrix and polymer matrix composites and engineering ceramics.
- Environmental effects due to in-service surface degradation by aggresive fluids or vapours, humidity and high temperatures.
- Detection and analysis of processing flaws in engineering ceramics and composite materials.
- Neutron diffraction measurements of internal stresses in metal matrix composite materials and in ceramic and metallic components.
- Detection and analysis of cracks in metals and ceramics and delamination in polymer composites.
- Enhancement of polymer processing by radiation modification of polymers.

3.1 Design with Polymer Composite Materials

Polymer composite materials have been used for advanced purposes within the aircraft and space industries for many years, and they are now becoming attractive for more conventional applications within different sectors of the man ufacturing industries. In order to be able to utilize them for still wider purposes, design data and design methods specific for these materials are needed. Design methodology and data for conventional materials are to a large extent based on both scientific research and experience from actual applications. Because of a lack of the latter for the broad range of these new materials, research aimed at establishing suitable data and design methods is performed in the Department.

When used for advanced load carrying structures, polymer composite materials are most often utilized with continuous fibres in the form of laminates, resulting in an anisotropic material with directionally dependent properties for both stiffness and strength. This has to be accounted for both in the analysis of the structure and in the evaluation, and several criteria must be applied simultaneously in the validation of a given component.

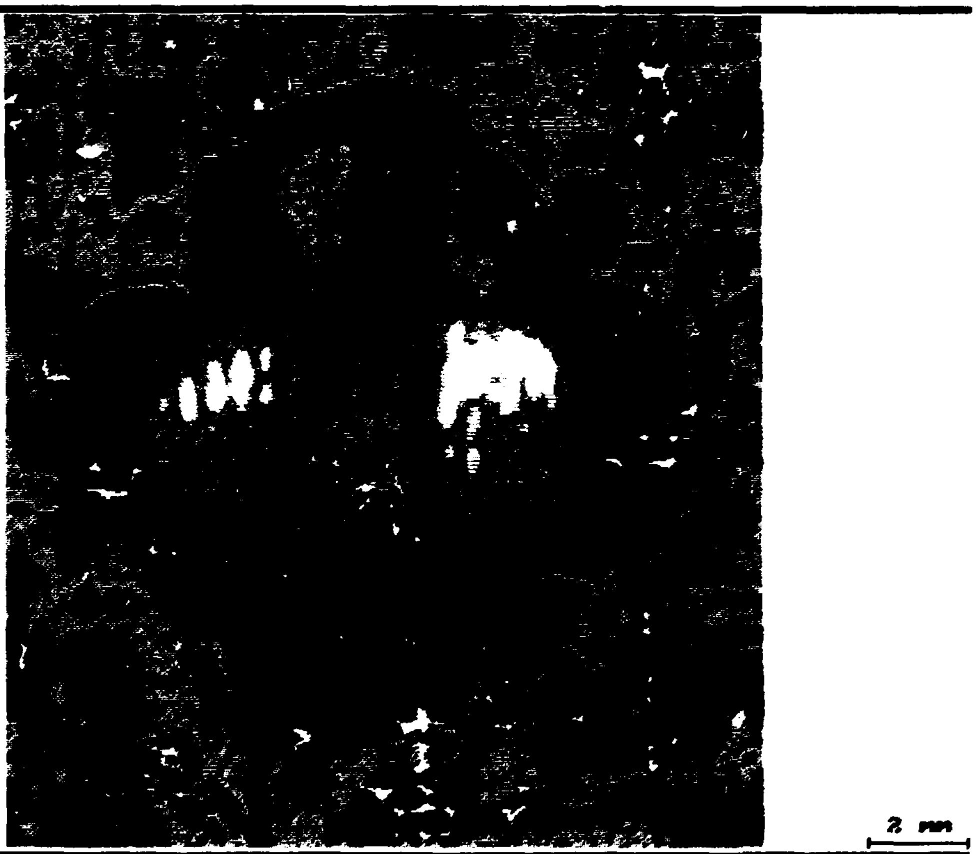
The design criteria may be based on either failure, deformations, stability or dynamic properties (eigenfrequencies), and the mechanical loads may be either stationary (static) or cyclic or dynamic (inertial effects, impact) and in principle any combination of these.

For stationary or static loading conditions, the structure can be designed either against failure from accidental overloading, where failure may be defined as total failure of one or all plies in a given section of the laminate, or it can be designed against cracks in the matrix material. This last criteria is considered conservative and it is supposed to give a safe design also against fatigue failure, but it may result in unnecessary use of material. In order to avoid this overdesign, suppression of the crack formation due to tensile stresses can be achieved by the introduction of compressive stresses in the matrix, and this design concept is investigated in an EURAM-project, in which the department is collaborating with MAN-Technology, Munich, FRG, the University of Napoli, Italy and CENTEC in Manchester, UK. In the project, compressive stresses are introduced in filament wound thin-walled cylindrical tubes (carbon/ epoxy), and the initiation of crack formation is studied and related to the level of initial pre- strain. The pre-stress concept is obviously easy to establish for an axisymmetric structure such as a tube, but it may be applicable to other components as well. It is important to assure that the beneficial effect of the compressive stresses are present throughout the whole lifetime of the component, implying that creep effects in the matrix material have to be taken into account.

Mechanical components are often exposed to cyclic loading, resulting in fatigue damage in the material. Until further understanding of the damage mechanisms in polymer composites is obtained, fatigue diagrams for the actual applied material in a component has to be available. For a class of materials - angle-ply glass/polyester - uniaxial fatigue tests have been performed as described in Section 2.5. The relative performance of two different ply-configurations for a given application depends on whether the loads are deformation (strain) controlled or load (stress) controlled. The designer may also have to take into account the relative stiffness reduction during the lifetime of the components, again depending on the specific application and component.

Local damage in polymer composite laminates may be caused by impact due to dropped items or collision with items. Depending on the degree of damage, the load carrying capacity and the stability against further fatigue damage may be reduced. Composite laminates are generally more sensitive to local loads and impact than metals. In an attempt to quantify the damage due to a local load, models for calculating the stress distribution under a localized load in a laminate are established, and tests on thick laminates are performed. In actual structures, evaluation of the consequences of a certain damage arises regarding the repair of damage and the properties of the repaired material. This problem is addressed in a new BRITE-program, where the Department is collaborating with three European helicopter manufacturers (Agusta, Italy; Westland, UK; MBB, Germany) and the University of Napoli, Italy. Different types of carbon/epoxy laminates and sandwich panels will be tested in the undamaged, the damaged and in the repaired state.

High resolution ultrasonic scanning image (C-scan) of indentation damage to a 9-layer cross-ply carbon/epoxy composite. Delaminations are detected between the upper three layers of the composite.



3.2 Testing of Polymer Composites

Testing of polymer composites with continuous fibres differs from testing of isotropic materials such as steel and alloys. The strength and elastic or inelastic properties are anisotropic and the thickness of the materials is often very small compared to other dimensions. Failure occurs progressively with the development of many cracks, primarily in the matrix material. These features are taken into account in current testing activities in the Department.

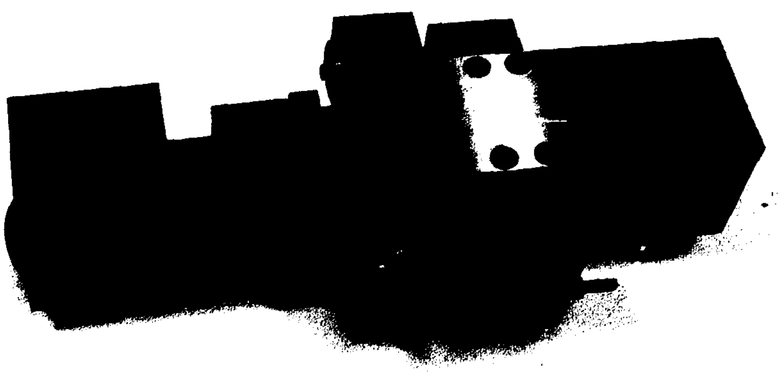
Interlaminar Fracture Toughness

The interlaminar fracture strength of polymer composites with carbon fibres in a thermohardening and a thermoset matrix can be characterized by measuring the interlaminar fracture toughness G_I for a thin unidirectional specimen with an artificial starter crack. Standardized methods are under development within the framework of ESIS (European Structural Integrity Society, formerly EGF), and tests are performed in the Department as a contribution to this development. The tests involve monitoring crack length as a function of load and displacement. This is done currently with visual observation with a travelling microscope. Alternative methods for automated and continuous monitoring of the crack length such as the potential drop method or crack gauges are being investigated. Parallel to this, the present microscope measurements are being automated, preparing for a possible future application of image analysis.

Fatigue Testing

Fatigue properties of glass-polyester materials are investigated within the framework of wind-energy-programs as described in Chapter 2.5. The same type of specimens is used for all tests, including those involving compressive loading. As the nominal specimen thickness is 2 mm, structural instability in the form of buckling will occur even at moderate compressive stresses. In order to avoid this, an anti-buckling device has been designed and successful applied. The gauge part of the specimen is clamped between steel plates, supplied with oval openings which allow extensometers to be attached to the specimen and observation of the damage development during the fatigue testing. The steel plates are clamped to the specimen with low clamping pressures in order to minimize frictional heating due to the relative movements between specimen and the plates. The plates are free to move in the axial (load) direction, but otherwise guided relative to the stationary (upper) part of the testing material. The actuator (lower) part of the testing machine may have some clearance in the sealing for the actuator; in order to avoid relative transverse movements between the upper and lower grips of the specimen, rollers are engaged at the outer part of the steel plates. The device has been used for a number of tests.

Anti-buckling device used in the fatigue testing of glass/polyester polymer composites.



3.3 Corrosion Fatigue of Steels

A 4 year BRITE (»Basic Research in Industrial Technologies for Europe«) project supported by the Commission of the European Communities) has been concluded in 1990. The project was a collaborative research program entitled »The Effect of Surface Degradation on Fatigue and Fracture Behaviour« with the Materials Department, Risø and partners from Germany, France, England and Ireland. (Daimler-Benz, Frankfurt, FRG; GEC ALSTHOM, Marcoussis, France; GEC ALSTHOM, Leicester, England; Trinity College, Dublin, Ireland)

The objective of the project has been the qualitative description of the influence of corrosion pits on the fatigue behaviour of components. Also, fatigue crack initiation and growth from a pit has been simulated.

The fundamental mechanisms of pit formation were studied. For this purpose a 13% Cr steel for gas turbine blades and a 3.5% Ni steel for rotors were investigated. A quantitative description of the influence of pit dimension, pit distribution and pit growth kinetics on fatigue behaviour in air and in corrosive environments has been evaluated. Surface analysis was carried out for studying the basic mechanisms and sources for pit formation.

Mechanical tests were carried out on smooth and prepitted specimens in air, and on smooth specimens in corrosive (pitting) environments. Crack initiation tests for four point bend test specimens at various stress levels were established in order to obtain the influence of the surface degradations on the initiation of fatigue cracks. Fracture mechanics tests were carried out on compact tension specimens to obtain the influences on threshold values and on fatigue crack growth behaviour. S-N fatigue tests were performed to generate basic fatigue data in order to develop lifetime curves. S-N fatigue tests of components (turbine blades and shafts) were also performed to confirm the accuracy and applicability of the models on real components.

All data have been used to establish a lifetime prediction model based on two theoretical approaches, both being based on a linear elastic fracture mechanics (LEFM) defect assessment. The models show excellent agreement for the 13% Cr steel and a conservative prediction of the fatigue strength reduction for the 3.5% Ni rotor steel.

3.4 Brittle Fracture of Welded Joints

A local brittle zone (LBZ) in a material is defined as a region of reduced fracture toughness, as compared to the material in the surrounding areas. For example, in multi-pass fusion welding of steel plates, a LBZ may be encountered in the heat affected zone (HAZ) of the base plate adjacent to the weldment where the microstructure is very inhomogeneous due to the different thermal cycles experienced during welding. Brittle fracture initiated from such LBZ's may result in complete structural failure.

Our objective is to detect the HAZ LBZ's and determine their significance for structural integrity. Impact Charpy-V- notch and fracture mechanics CTOD tests are used. Finite element based analysis of fracture of inhomogeneous material was performed.

The programme, which is sponsored by the National Agency of Trade and Industry and the Nordic Industrial Foundation, is carried out as a collaboration between a number of Danish, Nordic and Japanese institutes, and it is to be concluded in 1991.

3.5 Fatigue Properties of Rapidly Solidified-Al Alloys

A BRITE/EURAM project to develop high strength aluminium transition metal base alloys produced by rapid solidification process has progressed by a major effort on experimental measurements of fatigue and tensile properties at room temperature and at elevated temperatures. The alloys are produced by partners in Tampere, Finland and in Trondheim, Norway. (Technical University of Tampere, Finland; Technical University of Trondheim, Norway; and Harwell, UK.) Mechanical testing, metallography and evaluation has been carried out by Risø and thermodynamics and microstructural investigations were done by Harwell, U.K.

Several promising alloys based on Al-8Fe have been selected and on-going work is aimed to quantify the structures and properties of these alloys. The mechancal properties measured are tensile properties and S-N fatigue behaviour at room temperature, 150°C and 275°C.

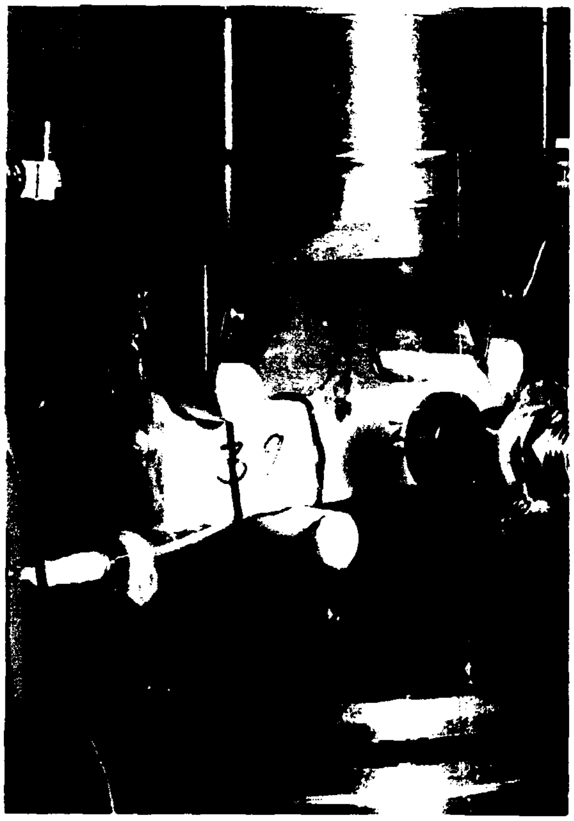
3.6 Mechanical Testing

The mechanical testing laboratory of the Materials Department also perform a variety of minor commercial projects for Danish industry and other institutions.

A contract on instrumenting polyethylene tubes for sewers in order to measure the strain and deformation during renovation of old cement sewer pipes was fulfilled. The renovation is carried out by shooting a new polyethylene tube through the old sewer and hereby replacing lengths of up to 100 metres without extensive digging work. During this relining the tube will be exposed to loads and deformations, which can reduce the lifetime of the new sewer. In-situ strain measurements were carried out during the relining process. This project was carried out in collaboration with The Danish Technological Institute, VBB in Sweden and the Danish contractor company, Hans Jørgensen & Son.

Other types of projects offered on a consultant and service basis are creep testing, tensile testing at high and low temperatures, fatigue testing, fracture mechanics testing, failure analysis and microstructural investigations.

Strain gauges are applied to the inside surface of high density polyethylene sewer pipes. The deformation in these pipes is monitored »in the field« during the relining of old cement pipes.



Measurement of fatigue crack initiation in a corrosive environment.

3.7 Testing of Engineering Ceramics

Research and development on engineering ceramics was mainly carried out within the »Centre of Advanced Technical Ceramics« which was established in spring 1989 within the Danish Materials Technology Development Programme. In this center, which is managed by one of the staff members of the Department, the research and development in the ceramics field is carried out in close collaboration with six major Danish industries and three research institutions. The Materials Department is engaged in the following centre projects: a) processing, science and technology for oxide- based ceramics; b) testing of engineering ceramics for specific applications; c) information and education. Besides these projects the Department is also engaged in projects on the development of ceramic gas sensors.

Wear Resistance

A wide range of ceramic materials has been characterized with respect to mechanical properties within the Centre for Advanced Technical Ceramics. The aim of the centre project is to find the most suitable ceramic material for seal rings used in water pumps. The abrasive wear resistance was investigated thoroughly. For this purpose a newly developed abrasive wear test, *Micro Wear Test*, was applied. The materials characterized were zirconia (ZrO_2), alumina (Al_2O_3), silicon nitride (Si_3N_4) and silicon carbide (SiC). For comparison, tungsten carbide hard-metal was also characterized. All of these materials are commercially available. Each material was tested at two different specific pressures, 500 and 1000 kPa, and against two different abrasives, quartz and silicon carbide. A general conclusion from the abrasive wear testing of these materials is that the most wear resistant ceramics are SiC and ZrO_2. If these materials are compared with the hard-metal, there is only a slight difference.

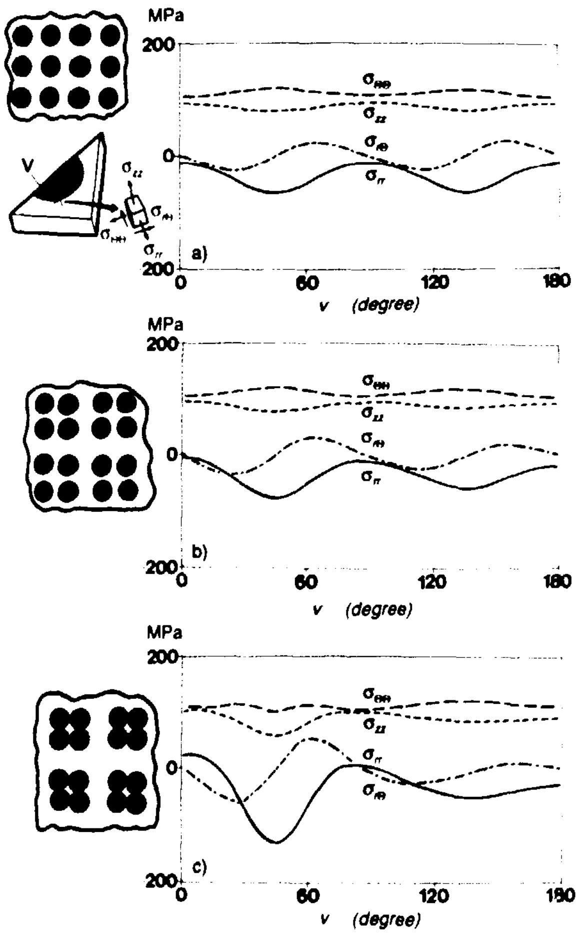
Fracture Toughness Measurements

There are several methods for determining the fracture toughness of a ceramic material. In the Materials Department two methods are used. One is the indentation fracture technique (IF) which is based on the traditional Vickers indentation for determining hardness. The only difference is that a sufficient load is applied so that cracks initiate in the corners of the indentation. The second method is newly developed and originated from the notch beam in four point bending technique. The traditional notched beam (NB) testing method is modified so that loading is controlled by the crack opening rate using a clip gauge. This system makes it possible to obtain slow crack growth in the ceramic materials. It is also possible to monitor the fracture toughness as a function of the crack extension, which gives the R-curve. This method has been applied to different types of ZrO_2. The results from these tests show that the fracture toughness values from the new method are similar to the traditional notched beam values. In comparison with the Vickers identation technique, the new method generally gives lower values.

Thermomechanical Properties of Ceramic Fibre Composites

The residual stress state in ceramic fibre composites strongly influences the overall mechanical behaviour (e.g. debonding, fibre pullout, thermal induced microcracking). The residual stresses exist due to thermal-elastic mismatch between fibres and matrix. This was investigated by 3 dimensional Finite Element Analysis. Various non-uniform fibre distributions were investigated (e.g. local fibre touching, fibre and matrix rich zones). It was found that the residual stress state was indeed sensitive to non-uniform fibre dispersion. Also, possible thermal damage modes were identified. This is being further investigated by thermal cycling experiments on a SiC-fibre glass matrix composite, using calcium aluminium silicate glass.

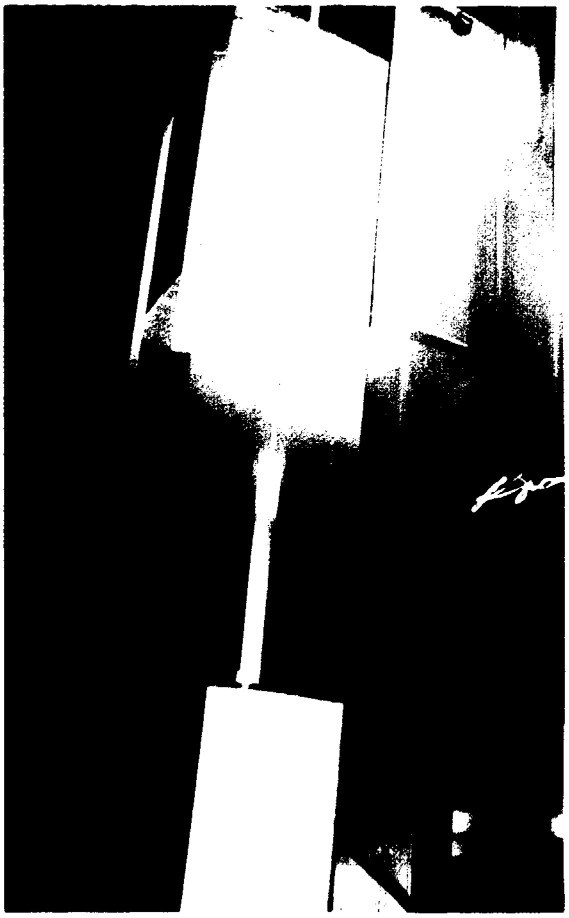
Residual stresses around fibres in SiC/CAS. a) perfect square array packing, b) four fibres lying close to each other, c) four fibres touching each other.



Thermal Shock Resistance

Techniques have been developed for testing of the thermal shock resistance of engineering ceramics. Besides the standard technique of quenching from fixed temperature levels into either boiling water or air at room temperature a technique involving thermal cycling between two temperatures was also developed. This latter technique was used to test the thermal shock resistance of commercial zirconia tubes for oxygen sensors. With the set-up shown in the figure below a cycling frequency of 18 times per hour between 500 and 1000°C with a maximum heating and cooling rate of 69 and 47°C/s respectively was obtained. The tubes were cycled 3000 times and most of the tubes tested could withstand this treatment without failure.

Thermal shock testing of zirconia tubes for oxygen sensors. The tube is repeatedly heated ~ 1000°C and then shock cooled as the furnace is moved up and down over the specimen.



Sensor Ceramics

Research was continued on development and testing of SO_2 gas sensors based on NaSiCON-electrolytes. Correct and reliable measurements could be performed with these sensors provided both electrodes are covered with sodium sulphate and corrections made for any temperature difference between the electrodes. Furthermore, two new types of sensors were developed by which measurements can be performed without the presence of a reference gas. The electrolytes used in these sensors were Na-β"-alumina and Ag-β"-alumina, respectively. In the first type the measuring gas is applied to both electrodes and the EMF is generated due to the temperature difference between the two electrodes. In the second type, which is selective towards SO_2, silver is used as a solid reference material.

3.8 High Resolution Ultrasonic Testing of Advanced Materials

During 1990, new ultrasonic equipment (HFUS 2000) and a step motor-driven scanning system has been installed. The higher measuring frequency (over 50 MHz instead of 25 MHz) made possible by the HFUS 2000 has improved the resolution in ultrasonic inspection considerably.

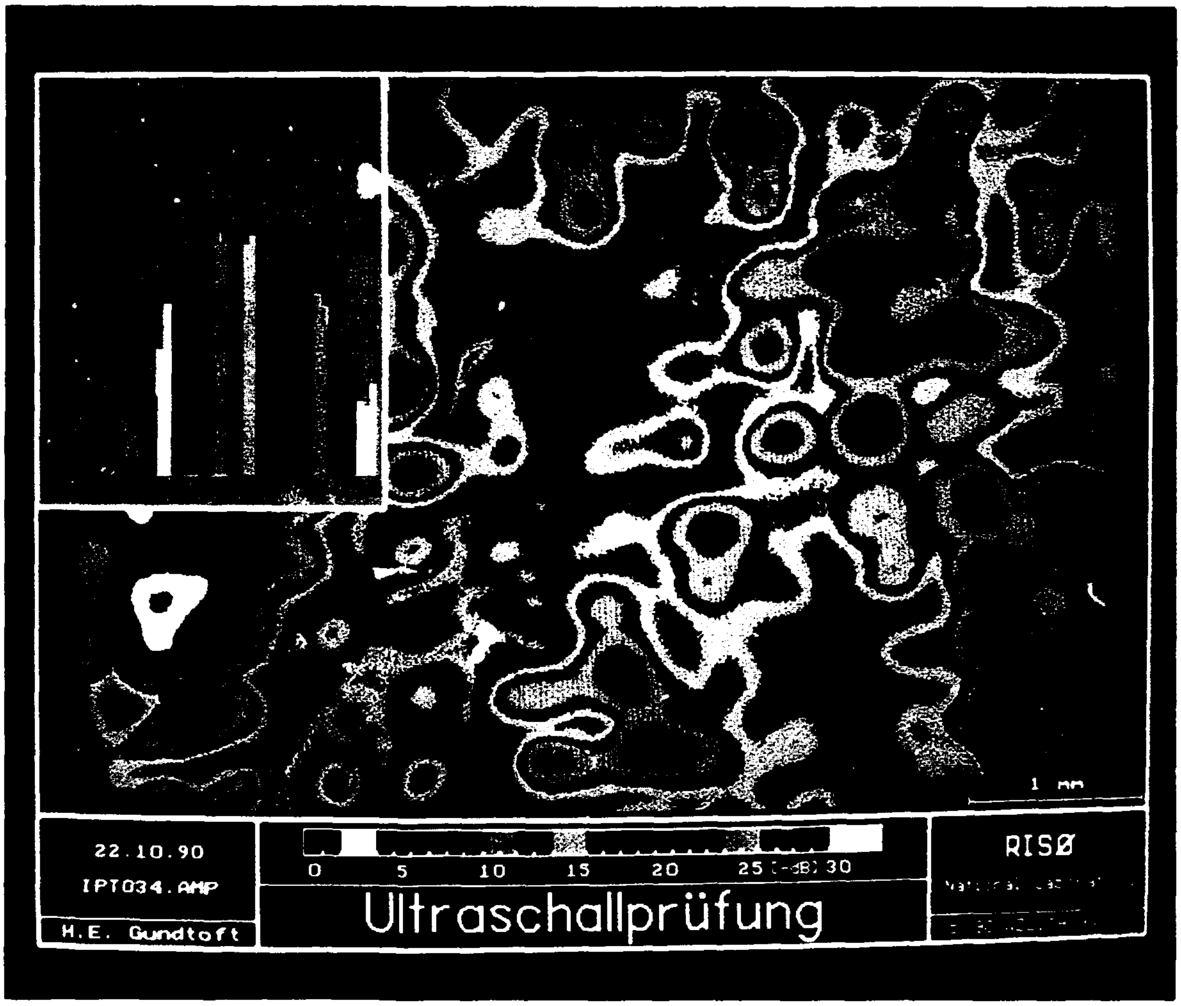
The complete system was delivered with software for measurement, data evaluation and presentation. During the year the new possibility offered with this system has been heavily used for scanning and characterisation of several advanced materials. Samples come from the two different groups of materials:

- composite (plastic or ceramic) and
- engineering ceramics.

Metals or powder metallurgical products can also be examined; our main projects are, however, within the two mentioned material groups.

Scanning can be performed in a hydraulic scanning system or a stepper-motor system. Sample size as well as data structure and inspection criteria differ between the two systems.

During 1990, our own software for the hydraulic scanning system has been under development. Practical scanning of the samples has therefore mostly been made in the stepper-motor scanning



High resolution ultrasonic · m of an area 6 × 4 mm in a 1 mm thick carbon/epoxy composite sheet.

system. The figure above shows a series of scannings of composite material with increasing resolution between the scans. In the highest resolution there is 0.01 mm between the scanning values. During one scan we can at the same time measure 2 echoheights and one distance between echoes. The picture shows heights of echoes from reflection in the material from delamination and pores. A histogram distribution of all measurements is also shown. The echoes can be chosen rather freely giving a lot of possibilities for finding internal defects or structural differences. Our experience from many scans during the year has shown several advantages and some shortcomings of the new steppermotor driven scanning system and software. This is illustrated by the following examples.

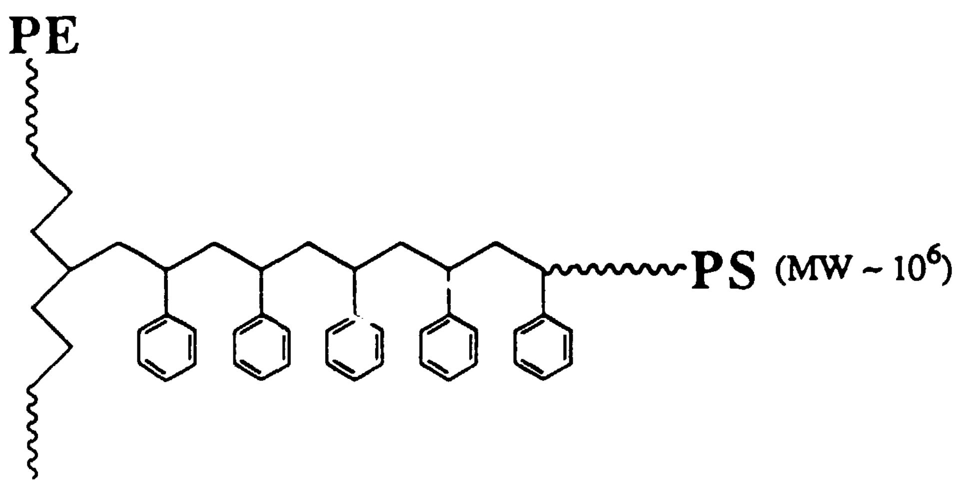
Scanning area can not be chosen freely but is linked to the resolution of the computer-screen. The system always stores mean values. Colours cannot be chosen freely. The scanning pattern is connected to the resolution of the computer screen in an inflexible way. Measurements are stored for all scanning positions and only mean values can be stored. Thus the system is not appropriate for finding single defects in ceramics. The hydraulic system is superior in this respect. An aim is therefore to make the stepper-motor system more flexible.

3.9 Radiation Modification of Polymers

Reactive species are formed by the interaction of ionizing radiation with organic materials, e.g. monomers and polymers. Chain crosslinking or chain scission are dominating reactions during irradiation of polymers, whereas a polymerization process is initiated by irradiation of pure monomer or monomer in the presence of a polymer (graft polymerization). The lifetime of the reactive species is normally so short, that all reactions will cease when the irradiation is stopped. Radiation induced reactions can therefore be terminated at any degree of conversion in a well controlled way. This is significantly different from thermally induced reactions.

The experimental work has focussed on the study of lightly crosslinked polymers around the gel point where insoluble material is formed, the modification of molecular weight distribution of a degradable polymer by controlled chain scission giving enhanced processing- and performance properties of polymer blends. Also, the development of a film matrix (PEPS) for solid phase reactions by radiation induced grafting has been studied. Due to careful selection of the polymerization medium, extremely long polystyrene (PS) chains are attached to polyethylene (PE) chains by covalent bonds. The polystyrene chains are easily functionalized in organic solvents without loosing the physical form of the film. This material is at present used for peptide synthesis (see Section 2.8).

Radiation allows polystyrene chains to be attached to polyethylene film. A Polystyrene molecular weight (MW) of more than 1 million can be achieved. Such large MW cannot be reached by traditional techniques; radiation initiation of free radical grafting is combined with a suitable reaction medium.



4 Materials Technology – Fabrication and Processing

The manufacture of advanced materials components often requires new processing, fabrication and joining techniques. Pilot plant studies of the production of fibre reinforced polymer composites, fine-powder metallurgical components and thin ceramic layered structures demand the construction of specialized equipment. This research and development also provides test specimens of new advanced materials for other programmes of the Department. The research activities in Materials Technology involve the manufacture of components of polymer matrix composites, new polymeric systems, engineering ceramics, prototype solid oxide fuel cells and fine-powder metals. Brazing and bonding techniques are being applied to a variety of these materials. The research programmes are carried out partly within the three Danish centres, Advanced Technical Ceramics, Powder Metallurgy and Polymer Composites, and partly in collaboration with other Danish and European research organizations and industrial partners.

Current research activities are concentrated in the following areas:

- Manufacture of advanced polymer composites by a variety of filament winding and fibre pre-form methods.
- The fabrication and joining of thin ceramic layers and oxide electrodes for Solid Oxide Fuel Cells.
- Inert gas atomization to produce fine metallic powders of new aluminium and iron-based alloys.
- Advanced technical ceramics processing.
- Brazing and joining techniques for powder metallurgical steels, aluminium alloys, superalloys, ceramics and ceramics to metals.
- New polymeric systems.

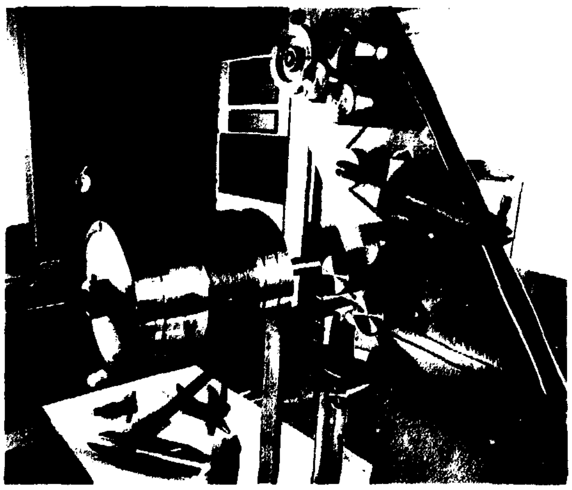
4.1 Manufacturing Processes for Advanced Composites

The involvement of the Materials Department in manufacturing of continuous fibre reinforced plastics serves mainly three purposes: a) the study of the fundamental principles of filament winding, autoclave processing, and resin transfer moulding, b) fabrication of test specimens, and c) development of prototype components.

The processing equipment consists of a computer-controlled filament winding machine, a hot-air high-temperature high-pressure autoclave, and equipment for resin transfer moulding.

A fundamental study of the filament winding technique has been completed. The influence of the process parameters (winding speed, fibre tension, temperature, and thickness of laminate) on the quality of wound laminates of fibre reinforced thermoset was examined. Three different winding processes were investigated: wet-filament winding where a wetting drum is immersed in a bath of matrix material; wet-filament winding where the matrix material is supplied in a quantity continuously controlled by the amount of fibre wound onto the mandrel; and dry-winding followed by a resin transfer technique. The

Filament winding of high temperature thermoplastic fibre composites. The wound specimens are subsequently consolidated in an autoclave process.



study was originally a confidential project, but the report has now been cleared, and the main conclusion from the study can be summarized as follows:

The maximum material quality, and that is; fiber content 60 vol%, porosity content less than 0.5 vol%, interlamina shear strength greater than 85 MPa. This was obtained by the following process parameters: wet-filament winding with continuously controlled supply of matrix material, winding speed of 16 m/min. (with a potential of even higher speed), fiber tension of 5 N, matrix viscosity of less than 30 mPa.s, obtained by a process temperature of 80°C for the actual matrix system.

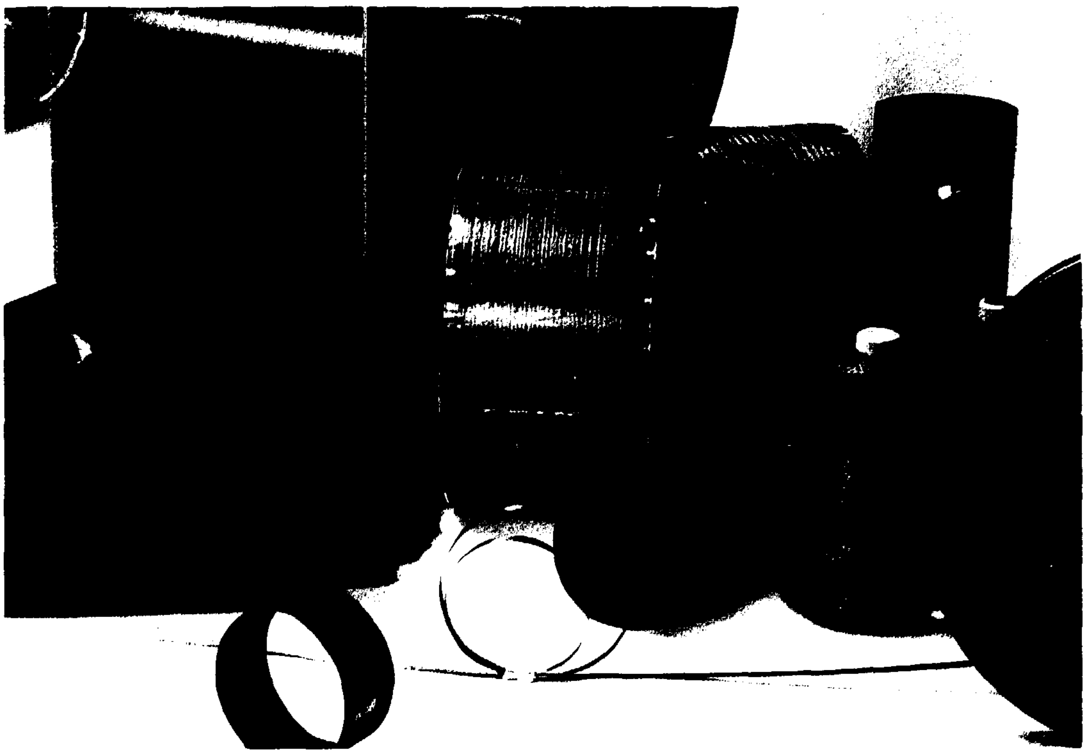
Two new projects on manufacturing technology for thermoplastic composites with continuous fibres were started late in 1989. The first project is on filament winding and the second one is on fibre pre-forms. (Fibre pre-forms are a pre-shaped fibre structure which has not yet been consolidated to the final structural component. Fibre pre-forms can be woven, knitted, braided or stitched together). The purpose of the projects is to examine the possibilities, limitations, and the characteristics of a fabrication technique where the material (fibre + matrix) is either wound onto a mandrel at room temperature or placed in a mould at room temperature in the form of a fibre pre-form, and consolidated by a subsequent autoclave process.

Initial results indicate that 235 mm diameter tubes of carbon fibre/PEEK can be processed (wound and consolidated) successfully if the wall thickness is not greater than 0.6 mm. At greater wall thicknesses there is a tendency that fibre wrinkles are formed at the outer surface of the tubes.

In the pre-form project the process technology for carbon fibre/PEEK has been established and the production of test panels is in progress. To illustrate the potential of this new technique a car door panel has been fabricated from woven cloth of carbon fibre/PEEK. The fiber composite door panel weighs only 25% of a similar steel door panel.

Another purpose of the filament winding project is to develop a computer aided design program (for use on a PC-computer) for filament wound structures. In the first step of this work the program will be limited to consider only geodesic paths on objects which are rotationally symmetric.

Hoop-wound tube specimens and plate specimens of carbon fibre reinforced thermoplastic, shown together with semi-raw material used for the fabrication of the specimens.

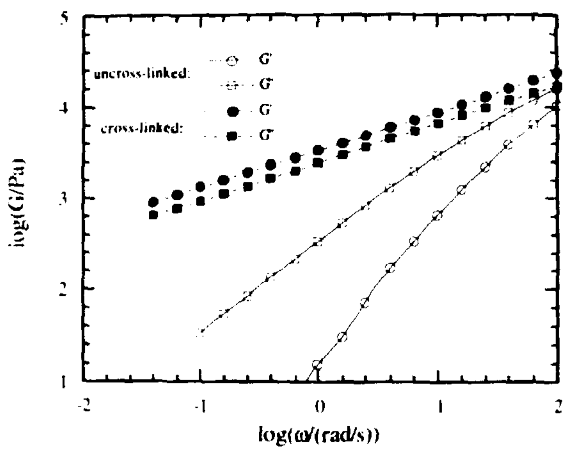


4.2 New Polymeric Systems

In the framework of the Materials Technology Development Programme a 3 year collaboration with Coloplast A/S, NKT A/S and Department of Chemical Engineering, Technical University of Denmark has been initiated. The collaboration has the exact title: »Polymeric Systems with Controlled Structure and Characteristics« is managed and coordinated by the Department.

The general goal of the program is to increase the competitiveness of that part of Danish industry that makes use of polymers. In this regard the concern is with processing, modification, and further development of polymers and raw materials that are available in order to manufacture improved products and comply with future (environmental) demands.

The primary task of the program is to shed light on the possibilities of producing materials and products with the most desirable characteristics fitted to the molecular- and microstructure of the selected materials. Morphology of polymer blends with controlled two-phase structure will be modelled with prediction of selected properties in mind. Through control and prognosis of the development of polymer adhesive mixtures, especially by means of crosslinking and adjustment of the rheological characteristics, adhesive mixtures are expected to be developed that have unique properties as adherents for plasters. Electrical characteristics of polymer materials are sought improved by use of thermoplastic elastomers resulting from controlled mixing and processing or by regulated crosslinking of selected components. Finally, model polymers with controlled structures, well-defined block copolymers and polymers with terminal functionality of relevance for the above mentioned areas will be prepared and evaluated.



Dynamical mechanical testing results of an uncross-linked silicone oil and a lightly cross-linked silicone. The elastic modulus (G') and the loss modulus (G") undergo dramatic changes on cross-linking. Good tack and cohesive properties are obtained by the electron beam cross-linking of the silicone oil.

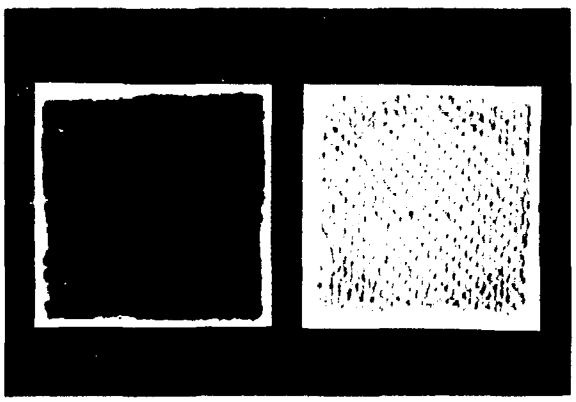
4.3 Solid Oxide Fuel Cells

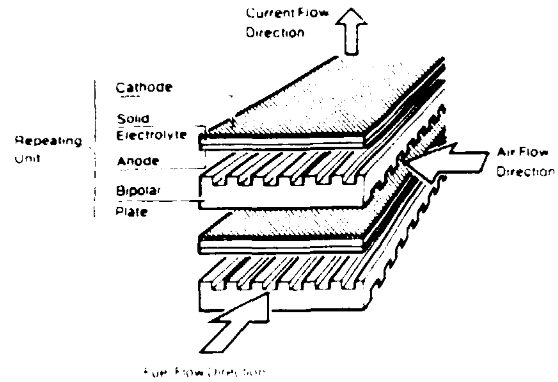
The Danish SOFC-related activities emerged from investigations at Risø of ion conducting inorganic compounds for solid state, secondary batteries. The initial SOFC work on materials properties gradually became more technologically oriented, including development of manufacturing techniques for ceramic materials.

More recently, the Danish government decided to focus its fuel cell support on the SOFC concept. As a consequence, a joint development program was organized as part of the government's Energy Research Program. In addition, there is Danish participation in several international programs on SOFC.

The Danish SOFC program was initiated in 1990 as a collaboration between five government research laboratories: The Materials Department of Risø, The Chemical Institute of Odense University, The Physical Chemical Institute, Physics Laboratory 3, and the Chemical Laboratory A, all of the Danish Technical University, and two industrial companies: Innovision (ERL) A/S and Haldor Topsøe A/S. The project management and coordination is in the Department. External support for the Danish SOFC program comes from the Danish government's Energy Research Program and the two groups of electric utilities ELKRAFT and ELSAM.

SOFC cell after testing, showing the air electrode side (black) and fuel electrode side (grey/green).





Schematic of the Risø SOFC fuel cell. The cell is made entirely of ceramic materials.

Activities

The main, current activities in the program are:

(a) Development of small cells and stacks, based on a flat-plate design and with »classical« materials: electrolyte YSZ (yttria stabilized zirconia), cathode (air electrode): $La(Sr)MnO_3$, anode (fuel electrode): Ni-YSZ, interconnect: $La(Sr)CrO_n$. A variety of techniques for obtaining the thin ceramic layers are examined, such as tape casting, extrusion, slurry coating, and screen printing. Cells and stacks will be tested with hydrogen.

(b) Investigation of new electroceramics which will enable direct conversion of natural gas and provide materials with improved ion and electron conductivity.

(c) Adaption of cell technology and material development to obtain experimental cells and stacks with improved materials. Cell testing will take place with methane/natural gas.

Achievements

The efforts in 1990 resulted in a number of achievements within the Danish SOFC programme. These achievements are listed below.

(i) Development of methods for the manufacture of »100g-to-kg« batches of suitable powder materials for cathode and interconnect (zirconia and nickel materials for electrolyte and anode are commercially available).

(ii) Establishing of reproducible tape casting procedures for the electrolyte for small cells, e.g. 4 x 4 cm^2.

(iii) Application of electrodes by slurry coating and serigraphy.

(iv) Preparation and testing (days-weeks) of single cells.

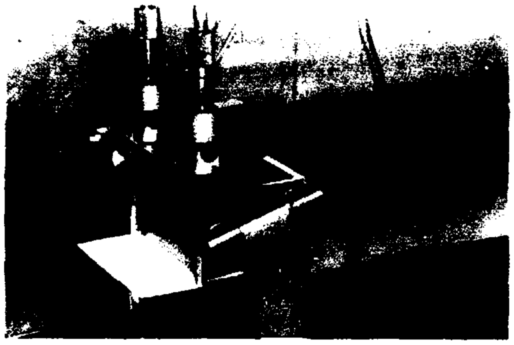
(v) Start with alternative shaping methods: extrusion of electrolyte, tape casting of electrodes, co- sintering of electrolyte and electrodes.

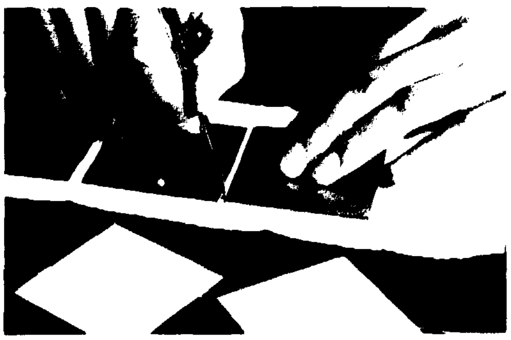
(vi) Promising results with new anode materials intended for direct methane conversion.

(vii) Initiation of long range research, partly on electric transport properties and stoichiometrics of cathode materials and partly on new methods for powder synthesis.

(viii) Participation in international collaboration (see below).

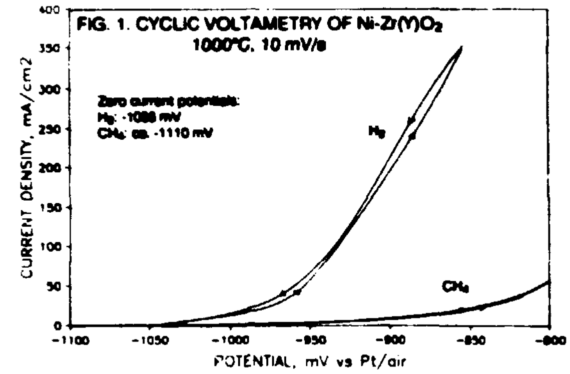
Various stages of the tape casting process are illustrated by the series of photographs to the right. Some of the interesting results with new anode materials are summarized in the following section.

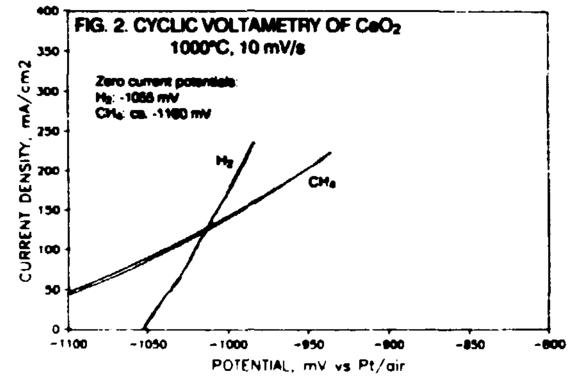




New Anode Materials

The Ni-YSZ anode has been tested at 1000°C with both H_2 and CH_4. As illustrated in the figure to the right, the oxidation rate (current density for CH_4 is more than a factor of 10 lower than for H_2 at a given potential. Previous work in the Department has shown that it is possible to oxidize CH_4 on a CeO_2-Gd_2O_3 type of electrode. A CeO_2 electrode with a proprietary additive can oxidize H_2 at a rate similar to that of Ni-YSZ, whereas the CH_4 oxidation rate can be 100 times higher.





Cyclic voltametry of anode materials for solid oxide fuel cells. The graphs show the results of tests at 1000°C using H_2 and CH_4.

International Collaboration

There is Danish participation in the following international SOFC-related programs:

- The »Nordic Tape Casting Project«, to advance the tape casting of ceramic materials. There are thirteen participants from the five Nordic countries. In addition to contributions from participants and national bodies, this project is supported by the Nordic Industry Fund. The Department participates on behalf of the Danish SOFC-group.
- The IEA-SOFC collaboration on »Modelling and Evaluation of Advanced SOFCs«. The Danish contribution will comprize the measurements of electrical transport properties, stoichiometrics, and internal strains by neutron diffraction. Also here, Risø participates on behalf of the Danish SOFC group.
- Two projects under the EC programs JOULE and BRITE/EURAM, to develop a 1 kW SOFC prototype. The projects are coordinated by ABB (FRG), other participants (besides Risø) are: ICI (UK) and TNO (NL), in JOULE also British Gas (UK) and SINTEF (N). The Department contributes to the following areas: tape casting, development of alternative materials, examination of microstructure/processing relationships.

4.4 Ceramic Processing

Materials research within the Centre project on processing science and technology was continued with the aim of developing new ceramic materials with improved fracture toughness. The research was concentrated on zirconia stabilized by ceria or magnesia as well as different types of ceramic composites.

Stabilized Zirconia Materials

For TZ12Ce (ceria stabilized zirconia containing 12 mole CeO_2) a coprecipitation technique was developed by which powders suitable for pressing and sintering of compacts of high density could be prepared. Flexural strength (determined by the 4-point bending technique) up to 525 MPa and fracture toughness values up to 40 MPa.m$^{1/2}$ were obtained on these compacts.

For MZ9 (magnesia stabilized zirconia containing 9 mol% MgO) the sintering conditions giving high strength and fracture toughness were established for a commercial powder (from Tosoh Corporation, Japan). These conditions were: firing at 1700°C for 3 hours followed by an isothermal anneal at 1340°C for 2 hours which gave a flexural strength of 450 MPa and a fracture toughness of 12 MPa.m$^{1/2}$ respectively.

Finally, partially stabilized zirconia was prepared by co-doping with magnesia and yttria. For materials containing about 9% mole equivalent Mg + Y a remarkable long term thermal stability at higher temperatures (1000°C) was observed which makes these materials interesting for oxygen sensor applications.

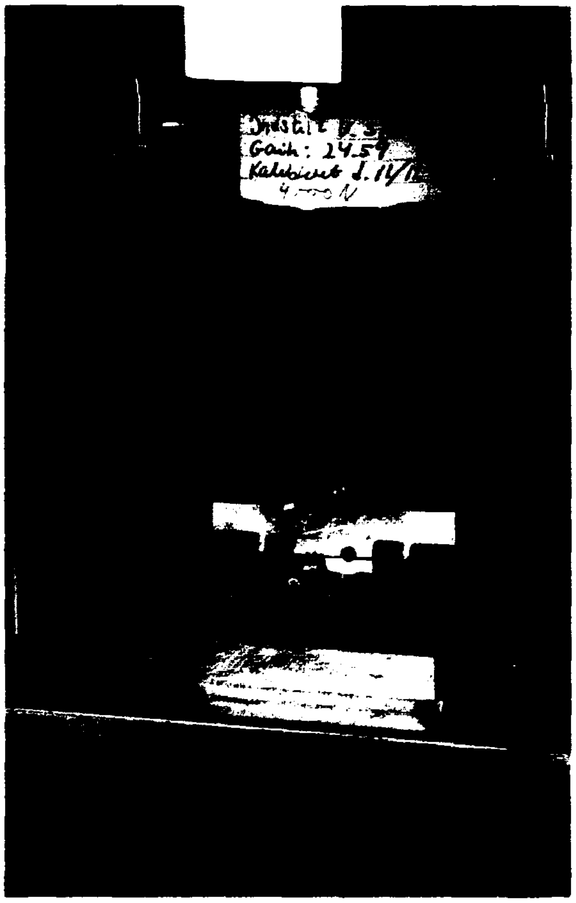
Ceramic Composite Materials

Ceramic-ceramic composites, consisting of a dispersed ceramic phase in a suitable ceramic matrix, offer the promise of improved mechanical properties over that of the matrix material alone. A significant increase in the corresponding fracture toughness can often be realized which offsets the strength degradation often seen in such materials. It is therefore necessary, through ceramic processing techniques, to minimize the strength loss while maximizing the fracture toughness of these composites.

A ZrO_2/20 wt% Al_2O_3 particulate composite was prepared by mixing the TZ12Ce powder described previously with a commercial Al_2O_3 (Alcoa A16) powder. Suspension mixing and ball milling were used to ensure a good dispersion of Al_2O_3 in the TZ12Ce matrix. Sintering of the composite powder at 1550°C for 2 hours resulted in a 98.5% dense material. A fracture toughness of 27.9 MPam$^{1/2}$ and flexural strength of 650 MPa were measured by Vickers indentation and the 4-point bend technique, respectively. The good mechanical properties and high density of the sintered composite resulted from the homogeneous dispersion of the reinforcing particulate (Al_2O_3) in the TZ12Ce matrix.

Short fibre or whiskers composites should have increased fracture toughness values relative to particulate composites since a greater number of toughening mechanisms are activated by their higher aspect ratios. Ceramic fibre composites were formed by incorporation of 10-20 wt% Al_2O_3 short fibres in a matrix of commercial tetragonal ZrO_2 containing 3 mol% Y_2O_3 (TZ3Y).

Mechanical testing of ceramic-ceramic composites.



The fibres were dispersed in a slightly basic solution and any agglomerated fibre bundles were removed by sedimentation. The fibres were then wet milled with the ZrO_2 powder and plastic balls to minimize fibre commutation. Pressureless sintering above 1500°C yielded densities of 99.5% and 99.8% for the 10 wt% and 20 wt% fibre materials, respectively. The 10 wt% fibre composite had a fracture toughness of 10.7 $MPa.m^{1/2}$ and a flexural strength of 750 MPa while the fracture toughness of the 20 wt% fibre composite was 10.0 $MPa.m^{1/2}$ and the bend strength 630 MPa. The use of wet processing techniques to eliminate the fibre bundles which act as stress concentrators was once again responsible for the good mechanical properties in this material.

Although ceramic whiskers and short fibres can increase the fracture toughness of a material, they may pose a potential health hazard during processing. A possible alternative for use in composite reinforcement is ceramic platelets. The intermediate aspect ratio of platelets make them environmentally safe while still allowing a large number of toughening mechanisms to operate. Alignment of platelets perpendicular to the advancing crack front would create the most favorable mechanical situation. Currently, research is being undertaken to align ceramic platelets within a ceramic matrix using a colloidal processing technique known as pressure filtration. Commercial yttria-stabilized ZrO_2 (TZ3Y) and single crystal Al_2O_3 platelets are dispersed in a liquid. A cake is consolidated at the filter interface and simultaneously the liquid is removed using applied pressure. This technique promises to be useful for formation of composites as well as single-component structural ceramic bodies.

Single crystal Al_2O_3 platelets for use as reinforcement in ZrO_2. Magnification × 10,000.

4.5 Powder Metallurgy Processing

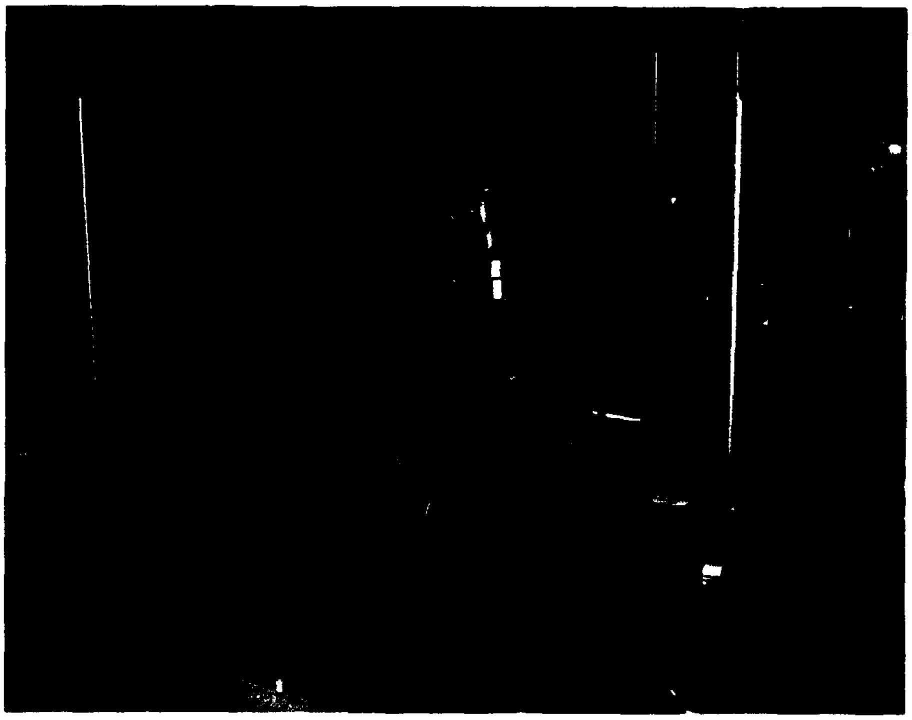
The Department's work in the powder metallurgical field has previously been concentrated on the processing of dispersion strengthened metals and metal matrix composites. This work has now been expanded into the areas of gas atomization and ultrafine powders. Both projects are part of the activities under the Centre for Powder Metallurgy within the Danish Materials Development Programme. The objective of these projects is to study the effect of various processing parameters on the characteristics of metal powders. Also, the mechanical, physical and chemical properties of consolidated material is being studied.

Gas Atomization

An inert gas atomizer for production of fine metal powders has been under construction during 1990 and is going to be finished during the first half of 1991. The atomizer is being constructed in collaboration with Niro Atomizer and the Danish Technological Institute.

In connection with the construction of the atomizer a risk analysis for metal dust explosions has been carried out. This analysis has formed an important part of the basis for the dimensioning of pressure loaded parts in case of explosion. Many passive precautions have been taken concerning dust explosion to prevent injuries to persons. The tank is equipped with two 4-inch overpressure reliefs (burst discs) breaking at approx. 3 bar. However because of the huge maximum rate of pressure rise for aluminium powder in the low-end particle size range (700 bar/sec. for 17 µm Al) burst discs are insufficient to ensure a safe venting of the system and therefore the entire equipment has been designed to withstand the explosion of the maximum amount of aluminium powder which could be found in the atomizer at any instant. Extended FEM-calculations have been done to document the ability of the tank to withstand the explosion under acceptance of plastic deformation.

Melting of the metals in the atomizer is done by a 25 kW induction heater supplied by a Danish manufacturer and is intended to cover the entire temperature range from a few hundred degrees centigrade and up to the melting point of iron.



Positron annihilation equipment for the investigation of fine powders in a controlled atmosphere at temperatures between -196°C and 500°C.

Ultrafine Powders

Ultrafine powders are being studied in collaboration with the Laboratory for Technical Physics, Technical University of Denmark.

Because of the extremely small particle size, 5 - 10 nm, a considerable part of the atoms are very close to a surface or a grain boundary. This is the reason for some unusual properties shown by ultrafine powders, like a very strong tendency to sinter and thereby minimize the total surface free energy. The affinity to sintering could be utilized for joining purposes (the joining of powder metallurgical parts).

Until now, ultrafine Fe/Ni powders of various compositions have been produced by the evaporation/condensation technique and characterized by a number of methods. In particular the reduction and oxidation behaviour has been analysed by micrograyimetric methods.

The experiments have shown that by oxidation of the homogeneous alloy, even at low (room) temperatures, the Fe segregates and oxidizes to form a mixed oxide of FeO and Fe_2O_3, whereas the Ni is left in its elemental form. During the following reduction in hydrogen at 100°C a complete reaction is obtained in a few hours and the homogeneous alloy of the initial composition is recovered.

In addition to the above mentioned measurements, equipment for positron annihilation experiments on ultrafine powder in hydrogen has been constructed and is now finished. Experiments will start early in 1991.

Manufacturing of Low Enriched Uranium Fuel Elements

Fabrication of LEU fuel elements for the Danish DR3 reactor was continued in 1990. The reactor has now totally converted to this new type of elements containing low-enriched uranium (LEU) with less than 20% U_{235}.

In addition to the low enrichment, these elements are special in the way the uranium is distributed in the rolled fuel plates. Cores containing the uranium are made powder metallurgically from a mixture of U_3Si_2 and Al-powder, before encapsulation in aluminium sheets.

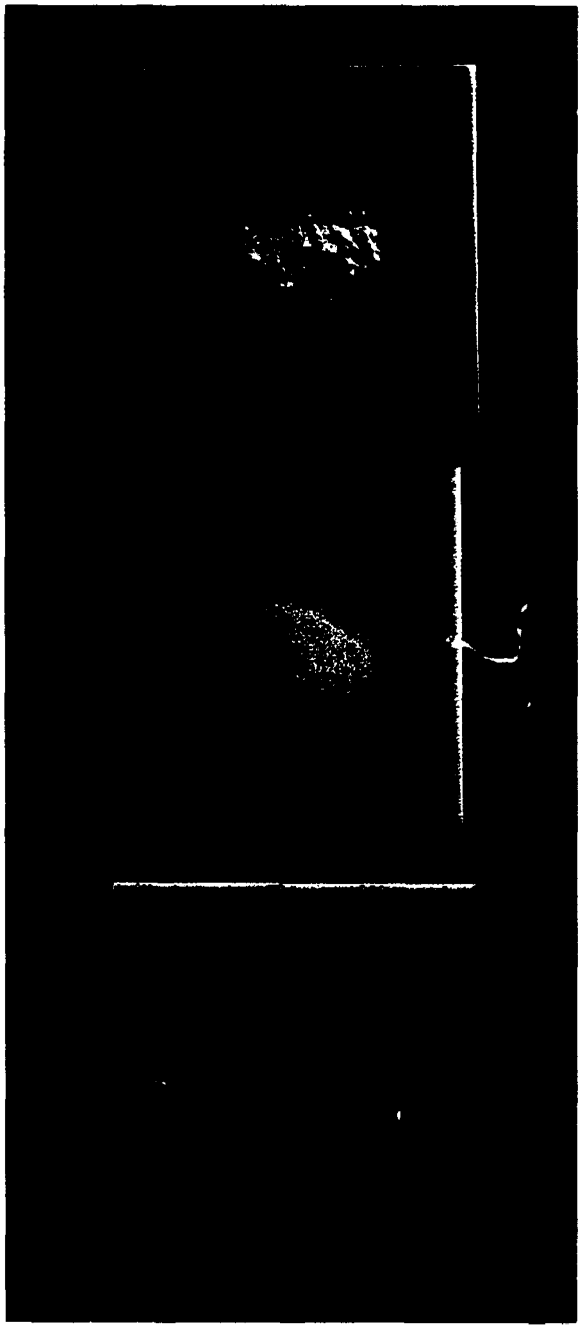
In 1990 a total of 45 elements was delivered to DR3.

4.6 Joining of Advanced Materials

An important part of the development of advanced materials is to study how such materials can be joined. Joining projects are therefore an integral part of the Danish Centres of Powder Metallurgy and Advanced Technical Ceramics. Part of the Department's work also includes the study of new brazing alloys which may be used in automated joining processes. Such developments are able to improve working conditions and cut costs, without reducing the quality of the finished products and components.

Brazing of Stainless Steels and Superalloys

The EEC-BRITE supported research programme on the development of improved new Ni-Cr-based brazing filler metals was continued in collaboration with the partners from FRG and the Netherlands. Extensive test programmes are carried out to determine the wetting-, tensile-, and fatigue properties. Also the corrosion resistance in dilute HNO_3 solution at 75°C has been investigated on the brazed joints. After having examined four selected commercial filler metals - one from each of the types - P, Si, B and B plus Si, a new series of braze alloys has been composed. Joints brazed with some of the new brazing alloys have shown both a lower and narrower melting range than the commercial ones.

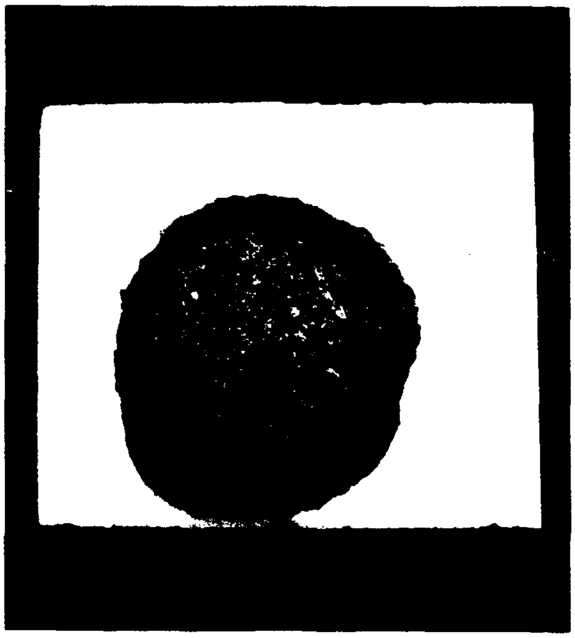


Wetting test specimens of new Ni-Cr-based brazing alloys. The examples shown are just three of a large test matrix covering brazing temperature as well as filler metal and substrate compositions.

Joining Ceramics

In the Danish Centre of Advanced Technical Ceramics the joining programme was continued. Investigations on brazed joints in PS ZrO_2 (3 mol-percent Y_2O_3) was continued. Using active brazing filler metals e.g. 70 Ag, 27 Cu, 3 Ti it was found that the 4-point bending strength was very much influenced by brazing temperatures between 800°C and 950°C as well as a cleaning procedure of the as-cut surface by firing in air at 1400°C.

Diffusion joining of Al_2O_3 was also continued. A more elaborate diffusion joining equipment - 1500°C max. and a load of 30 kp - has been completed. Several vacuum tight tubes closed by Al_2O_3/ZrO_2 joints using a Pt foil as interlayer have been produced. The bonded closed tubes have been tested by thermal cycling between 200 and 800°C showing no leaks after more than 1000 cycles.



Wetting of ceramic test specimen by an active brazing filler metal containing 3% titanium.

Joining P/M Materials

In the Danish Centre for Powder Metallurgy the joining programme was continued and experimental work has started on brazing and diffusion bonding of sintered P/M-parts.

In this work the diffusion bonding process has shown the best results so far, as solid state diffusion bonding creates no difficulties with respect to the porosities in the powder material. Diffusion bonding has been carried out on materials based on steel and stainless steel powders under conditions similar to those of sintering these materials. So far, sintered and calibrated P/M-parts have been bonded; strengths of approx. 80% of the parent materials have been achieved with a load corresponding to a nominal contact pressure during bonding of 3 MPa (max. obtainable).

To obtain a higher contact pressure, a new diffusion bonding facility has been designed and installed in a vacuum furnace. Maximum bonding parameters are 1200°C, 60 kN and specimen dimension 50 mm × 50 mm × 150 mm.

Brazing techniques for brazing porous materials are being studied. Experimental work is carried out using conventional brazing methods and especially in the field of transient liquid phase brazing.

5 Participation in Danish and International Committees

The Department is widely represented in scientific, educational and technical committees within Denmark and abroad. Such participation provides a base for international collaborative research activities and information exchange. The responsible staff member may be contacted for further information on activities within a particular area.

International

Council of the European Peptide Society.
Rolf H. Berg.

The European Structural Integrity Society. Delft, The Netherlands.
Christian Debel, Povl Brøndsted.

The European Neutron Radiography Working Group. Joint Research Centre. Petten, Belgium.
Jozef C. Domanus.

The International Advisory Committee on »International Conferences on Positron Annihilation«.
Morten Eldrup.

The Halden Programme Group, OECD.
Niels Hansen.

The COST 501 Management Committee on Materials for Energy Conversion Using Fossil Fuels. Brussels, Belgium.
Niels Hansen.

The Fusion Technology Steering Committee. EEC. Brussels, Belgium.
Niels Hansen.

The Editorial Board of »Journal of Nuclear Materials«.
Niels Hansen.

The Editorial Board of »Monographs in Materials Science«.
Niels Hansen.

Expert Group on Structural Materials, Fusion Technology Programme. EEC, Brussels, Belgium.
Niels Hansen (Chairman), Bachu N. Singh.

European Polymer Federation. Soronto, Italy.
Søren Hvilsted.

The IAEA International Working Group on Water Reactor Fuel Performance and Technology. Vienna, Austria.
Per Knudsen (Chairman).

The Editorial Advisory Board of »Textures and Microstructures«.
Torben Leffers.

The Organizing Committee for NATO Advanced Research Workshop on »Measurement of Residual and Applied Stress Using Neutron Diffraction.« Oxford, UK.
Torben Leffers.

The International Scientific Committee for the 5th International MECAMAT Seminar on »Large Plastic Deformations, Fundamentals and Applications to Metals Forming«, Fontainebleau, France.
Torben Leffers.

The Editorial Board of »Composites Science and Technology«. London, UK.
Hans Lilholt.

International Committee for Composite Materials. Philadelphia, USA.
Hans Lilholt.

European Association for Composite Materials: Standing Committee. Bordeaux, France.
Hans Lilholt.

The Information Exchange Group under the European Space Agency on Carlson Fibre Reinforced Plastics, Nordweijk, The Netherlands.
Aage Lystrup.

Fuel Cell Research Group, Nordic Council of Ministers. Copenhagen, DK.
Finn Willy Poulsen.

International Society for Solid State Ionics, ISSI. Rome, Italy.
Finn Willy Poulsen (Councillor).

The Working Party on Nuclear Corrosion, The European Federation of Corrosion.
Kaj Rørbo.

The ISPRA Cyclotron Users Committee, Joint Reseach Centre. Ispra, Italy.
Bachu N. Singh.

Expert Group on Plasma Facing Components, Fusion Technology Programme, EEC. Brussels, Belgium.
Bachu N. Singh.

International Advisory Committee of »The 15th International Symposium on the Effects of Radiation on Materials«.
Bachu N. Singh.

European Society for Materials Education, Delft University. Delft, The Netherlands.
Ole Toft Sørensen.

Nordic Society for Thermal Analysis and Calorimetry, Risø National Laboratory, DK.
Ole Toft Sørensen (Chairman).

The Editorial Board of »Journal of Materials Education«. Pennsylvania State University, USA.
Ole Toft Sørensen.

The Editorial Board of »Thermochimica Acta«.
Ole Toft Sørensen.

Danish

The Advisory Group for Transport. Danish Ministry of Energy. Copenhagen.
Eivind Adolph.

Technical Assessors, Danish Institute of Fundamental Meteorology, Lyngby.
Eivind Adolph, Niels Hansen.

The Advisory Board of: »Materials Technology in the Danish Rubber Industry«. Materials Technology Development Programme.
Walther Batsberg Pedersen.

The Board of Governors of Risø National Laboratory. Roskilde.
Morten Eldrup, John Kjøller (Staff Representatives).

The Danish Ministry of Industry; Reference Group for the BRITE/EURAM programme. Copenhagen.
Niels Hansen.

The Advisory Group for Fuel Cells, Danish Ministry of Energy. Copenhagen.
Niels Hansen

The Steering Committee for Danish Oxide Fuel Cells Programme.
Niels Hansen

The Steering Committee for the Centre for Advanced Technical Ceramics.
Niels Hansen

The Advisory Committee for Materials Testing. The Danish Technological Institute, Aarhus.
Niels Hansen

The Executive Committee of the Danish Society for Polymer Technology. Copenhagen.
Søren Hvilsted (Chairman).

The Research Committee of the Danish Society of Chemical, Civil, Electrical, and Mechanical Engineers. Copenhagen.
Torben Leffers (Chairman).

The Danish Technical Research Council. Copenhagen.
Torben Leffers.

The Executive Committee of the Danish Metallurgical Society. Lyngby.
Povl Brøndsted.

The Steering Committee for the Centre for Polymeric Composite Materials. Copenhagen.
Hans Lilholt.

The Executive Committee of the Danish Electrochemical Society. Lyngby.
Finn Willy Poulsen (Vice Chairman).

6 Education and Training

Many of the Department's staff members are actively involved in education and training in materials science as university external lecturers and examiners. Further, research on projects by undergraduate, post-graduate and post-doctoral students was carried out under the supervision of Department staff members.

External Lecturers

Walther Batsberg Pedersen.
»Advanced Course on Liquid Chromatography«. Royal Danish School of Pharmacy, Copenhagen, DK.

Niels Hansen, Kaj Rørbo.
Materials Science. Danish Academy of Engineering, Lyngby, DK.

Kaj Rørbo, Povl Brøndsted.
»Improved Corrosion Evaluation and Protection«. UN course, held at Risø.

Jørgen B. Bilde-Sørensen, Kaj Rørbo.
Development Program, in collaboration with Haldor Topsøe A/S and the Danish Corrosion Centre.

Ole Toft Sørensen.
»Introduction to Non-stoichiometric Oxides/Solid State Chemistry«. Technical University of Denmark, Lyngby, DK.

External Examiners

Walther Batsberg Pedersen.
M.Sc. examiner, Technical University of Denmark, Lyngby, DK.

Ole Bøcker Pedersen.
Ph.D. examiner, University of Cambridge, UK.

Søren Hvilsted.
Ph.D. examiner, Technical University of Denmark, Lyngby, DK.

Hans Lilholt.
M.Sc. examiner, Technical University of Denmark, Lyngby, DK.

Hans Lilholt.
M.Sc. examiner, University of Aalborg, DK.

Finn Willy Poulsen.
Ph.D. examiner, University of Salford, UK.

Ole Toft Sørensen.
M.Sc. examiner, Technical University of Denmark, Lyngby, DK.

Ole Toft Sørensen.
M.Sc. examiner, University of Aalborg, DK.

Colloquia held at Risø

»Coarsening of Gas Bubbles in Solids«. 1 Mar.
Dr. Helmut Trinkaus, Forschungsanlage Jülich, FRG.

»The Influence of the Interannealing Treatment on the Deformation and Recrystallization Texture of Commercial Aluminium«. 9 Mar.
Dr. Adam Bunsch, Academy of Mining and Metallurgy, Krakow, Poland.

»Formation of Microbands in Rolled Al + 5.5% Mg«. 26 Mar.
Dr. Darcy A. Hughes, Sandia National Laboratories, Livermore, USA.

»Composites Research and Sol Techniques at Queen's University«. 24 Apr.
Prof. Reginald Smith, Queen's University, Kingston, Canada.

»Creep of the Intermetallic Compound TiAl«. 7 May.
Prof. H. Oikawa, Tohoku University, Japan.

»Materials and Processes in Solid Oxide Fuel Cells«. 14 May.
Dr. Mogens Mogensen, Risø National Laboratory.

»Cathode Materials and Solid Oxide Ion Conductors for Solid Oxide Fuel Cells«. 21 May.
Prof. O. Yamamoto, Mie University, Japan.

»Studies on Inorganic Sols«. 31 May.
Dr. Bruno Kindl, Queen's University, Kingston, Canada.

»Development Status of SOFC«. 2 July.
Professor S. Uchida, Mitsubishi Heavy Industries Ltd., Japan.

»Positron Annihilation Studies of Helium in Metals and Alloys: Recent Works at Kalpakkam«. 15 Aug.
Dr. B. Viswanathan, Indira Gandhi Centre for Atomic Research, Kalpakkam, India.

»Rolling Conditions for High R-Value of Hot Rolled Steel Sheets Analyzed by Computer Simulation Model for Microstructure«. 25 Sept.
Dr. T. Nakamura, Nippon Steel Corporation, Japan.

«Brainstorming: What can a Materials Scientist Usefully do with a Scanning Tunneling Microscope?«. 28 Sept.
Dr. Jørgen B. Bilde-Sørensen, Risø National Laboratory.

«Some Views on Plastic Deformation and the Grain Size Dependence of the Flow Stress«. 28 Nov.
Prof. Brendon Parker, Monash University. Clayton, Victoria, Australia.

Undergraduate Projects

The following students carried out project research work within the Department towards their degrees at universities within Denmark or abroad. The project title, student affiliation and Risø supervisors are given.

Pauline Buitink.
»Comparison of Different Conductivity Measurement Methods on YSZ-Tapes.« Technical University of Delft, NL.
Finn Willy Poulsen.

Christian Dræby and Jesper Højmark Esbensen.
»Metal-Matrix Composite: Al-SiC«.
Christian Debel.

Torben Elhauge.
»Application of Photoacoustic Spectroscopy for Studying Polymeric Composites«. Danish Academy of Engineering, Lyngby, DK.
Søren Hvilsted.

Richard W.A. Francis.
»Flow Stress Anisotropy in Cross Rolled Aluminium«. Brunel University, UK.
Dorte Juul Jensen.

Per Lyngsø Hansen.
»Ceramic Coatings made by Spray Pyrolysis« Danish Academy of Engineering, Lyngby, DK.
Finn Willy Poulsen.

Thomas Lindegaard and Uffe Rud Hansen.
»Ceramic Oxide as Anode Material in Solid Oxide Fuel Cells«. Danish Academy of Engineering, Lyngby, DK.
Mogens Mogensen.

Nelleke van der Puil.
»Synthesis and Characterization of La- and Sr-Zirconate« Technical University of Delft, NL.
Finn Willy Poulsen.

Post-graduate Projects

The following students carried out research work within the Department towards their Ph.D. degrees. The project title, student affiliation and Risø supervisors are given.

Charlotte Clausen.
»Characterization of Interfaces in Ceramics by Electron Microscopy«. Technical University of Denmark/Danish Research Academy.
Jørgen B. Bilde-Sørensen.

Kurt Heller.
»Rheological Studies of Polymer Solutions and Gels«. Roskilde University Centre.
Walther Batsberg Pedersen.

Ole Jørgensen.
»Impact Damage in Polymer Composites«. Technical University of Denmark/Danish Research Academy.
Svend Ib Andersen.

Torben Lorentzen.
»Measurements of Internal Residual Stresses by Neutron Diffraction«. Aalborg University Centre.
Dorte Juul Jensen and Torben Leffers.

Bent Sørensen.
»Ceramic Fibre Composites: Thermomechanical Behaviour.« Technical University of Denmark/Danish Research Academy.
Christian Debel and Ole Toft Sørensen.

Niels Jacob Sørensen.
»Thermomechanical Properties of Metal Matrix Composites at High Strains and Temperatures.« Technical University of Denmark/Danish Research Academy.
Niels Hansen, Dorte Juul Jensen and Hans Lilholt.

David Tricker.
»The Electron Microscopical and Electrical Characterization of Boundaries in Solid Oxide Fuel Cell Materials«. University of Cambridge, UK.
Jørgen B. Bilde-Sørensen.

7. Finances

The Department receives its financial support partly through basic governmental funding, and partly through national and international project oriented funds.

The numbers given are in units of 1000 Danish kr. In parantheses the equivalent number in units of 1000 US$ (100 DDK equals 15.5 US$) is given.

Income

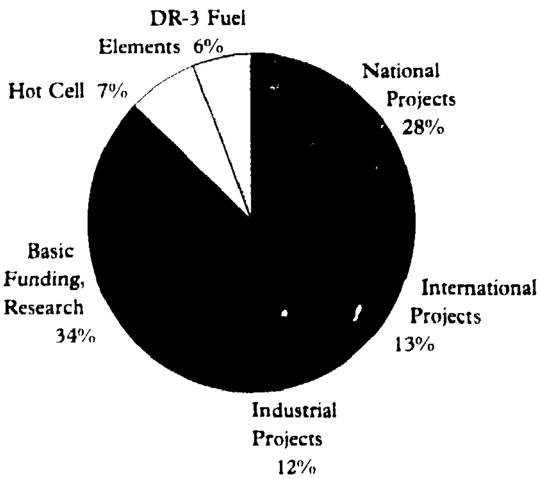
	DDK x 1000	$ x 100
Basic funding: (Ministry of Energy)	20450	3180
Project funding:	25200	3920
	45650	7100

Investments

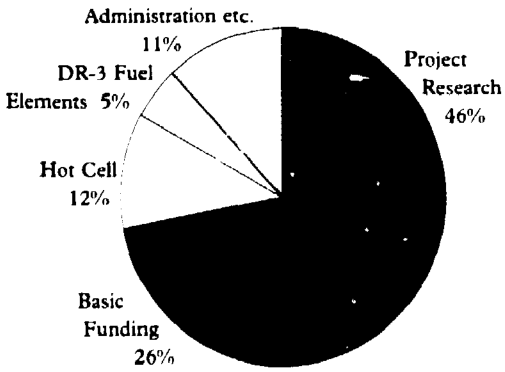
	1000 DDK	1000 US$
Equipment	3650	568
Financing:		
Basic Funds	200	31
Project Funds	3450	537

Expenditure 1990

	Basic Funds		Project Funds		Total	
	1000 DDK	1000 US$	1000 DDK	1000 US$	1000 DDK	1000 US$
Salary	16750	2605	9450	1470	26200	4075
Running expenses:	3500	544	6550	1019	10050	1563
Equipment:	200	31	3650	568	3850	599
Tax:	-	-	550	86	550	86
Administrative charges:	-	-	3600	560	3600	560
Total:	20450	3180	23800	3703	44250	6883



Distribution of income
(Total of 45.65 MDDK)



Manpower distribution
(Total 43 scientific, 54 technical and office staff)

8. Staff of the Department

Head of Department
Niels Hansen

Scientific Staff
Adolph, Eivind
Almdal, Kristoffer
Andersen, Svend I.
Bagger, Carsten
Bentzen, Janet J.
Berg, Rolf H.
Bilde-Sørensen, Jørgen B.
Borring, Jan
Borum, Kaj K.
Brøndsted, Povl
Bøcker Pedersen, Ole
Carlsen, Hans
Christensen, Jørgen
Debel, Christian P.
Eldrup, Morten M.
Gotthjælp, Klaus
Gundtoft, Hans E.
Horsewell, Andy
Hvilsted, Søren
Johansen, Bjørn S.
Juul Jensen, Dorte
Knudsen, Per
Leffers, Torben
Lilholt, Hans
Liu, Yi-lin
Lorentzen, Torben
Lystrup, Aage
Løgstrup Andersen, Tom
Malmgren-Hansen, Bjørn
Mogensen, Mogens B.
Møller, Carsten V.
Pedersen, Walther Batsberg
Poulsen, Finn W.
Rørbo, Kaj
Schrøder Pedersen, Allan
Singh, Bachu N.
Thomsen, Niels B.
Toft, Palle
Toft Sørensen, Ole
Toftegaard, Helmuth L.

Postgraduate Students
Clausen, Charlotte
Heller, Kurt
Jørgensen, Ole
Sørensen, Bent
Sørensen, Niels Jacob
Tricker, David

Guest Scientists
Ananthan, V.S.
Nyberg, Eric

Consultants
Domanus, Josef
Kindl, Bruno
Nilsson, Tage M.
Tetzschner, Mogens
Waagepetersen, Gaston

Technical Staff
Adrian, Frank
Andersen, Axel B.
Aukdal, Jørgen A.
Borchsenius, Jens F.S.
Bülow Christensen, Carl J.
Bønke Nielsen, Anne
Christensen, Svend E.
Dreves Nielsen, Poul
Frederiksen, Henning
Gravesen, Niels Nørgaard
Hansen, Lotte
Hansen, Niels J.
Hersbøll, Bent
Hubert, Lene
Jensen, Finn E.
Jensen, Knud
Jensen, Palle V.
Jepsen, Ole Kjær
Jespersen, John
Kjøller, John
Klitholm, Cliver
Larsen, Bent
Larsen, Jan
Larsen, Kjeld J.C.
Lindbo, Jørgen
Mikkelsen, Claus
Nielsen, Ove
Nielsen, Palle H.
Nielsen, Preben
Nilsson, Helmer
Olesen, Preben B.
Olsen, Benny F.
Olsen, Bent K.
Olsen, Henning
Olsen, Ole
Olsson, Jens O.
Paulsen, Henrik
Pedersen, Børge E.
Pedersen, Knud E.
Pedersen, Niels Jørgen
Petersen, Jan H.A.
Robl, Steen
Sandsted, Kjeld
Strauss, Torben R.
Sørensen, Erling
Thomsen, Leif F. T.
Aagesen, Sven

Office Staff
Dreves Nielsen, Elsa
Helbo Pedersen, Anne
Hoffmann Nielsen, Lis
Jørgensen Lone
Lauritsen, Grethe W.
Sørensen, Eva M.
Thomsen, Ann

Apprentices
Christensen, Kim
Enghave, Christian
Hansen, Martin
Jensen, Keld Lillegård
Madsen, Niels O.
Olsen, Morten
Westergaard, Kenneth B.

In 1990 the Polymer Group, comprising 4 scientific and 3 technical staff, was transferred to the Department.

9 Visiting Scientists and Students

The following guest scientists worked in the Department during 1990:

Dr. Brent Adams.
Yale University, USA. 18 June - 18 July.

Prof. Frank S. Bates.
University of Minnesota, USA. 14 - 22 Dec.

Dr. Claire Barlow.
University of Cambridge, UK. 27 Aug. - 27 Sept.

Dr. Adam Bunsch.
Instytut Metalurgii, Akademia Gornicza-Hutnicza, Krakow, Poland. 1 Dec. 1989 - 1 Apr. 1990.

Dr. Didier Gavillet.
Paul Scherrer Institute, Villigen, Switzerland. 26 - 31 Aug.

Dr. Xuewu Ge.
University of Science and Technology of China, Hefei, Anhui. 15 Mar. - 31 Aug.

Dr. Howard L. Heinisch.
Battelle Pacific Northwest Laboratory, Richland, USA. 22 Oct. - 4 Nov.

Dr. Darcy A. Hughes.
Sandia National Laboratory, USA. 24 Feb. - 27 Mar.

Dr. Andy R. Jones.
AEA Technology, Risley, UK. 16 - 27 July.

Prof. Ding-kun, Peng
University of Science and Technology of China, The People's Republic of China. 1 Jan. - 1 Apr.

Prof. Brian Ralph.
Brunel University, UK. 16 - 20 May.

Dr Abdoul Saoucha.
Haut Commissariat a la Recherche, Centre de Development des Techniques Nucleaires, Alger, Algeria. 1 Mar. - 28 Apr.

Dr. Walt F. Sommer.
Los Alamos National Laboratory, Los Alamos, USA. 15 Jul. - 1 Aug.

Dr. Helmut Trinkaus
KFA, Jülich, FRG. 28 Feb. - 4 Mar. & 25 - 28 Aug.

Dr. Max Victoria
Paul Scherrer Institute, Villigen, Switzerland. 22 - 27 Apr.

Dr. Phil J. Withers
University of Cambridge, UK. 13 Aug. - 31 Aug.

Dr. Chung H. Woo.
Atomic Energy of Canada Ltd., Pinawa, Canada. 15 - 27 Oct.

The following foreign students (post-graduate) worked in the Department during 1990:

Arup Basu.
University of Manchester, UK. 16 - 30 Nov.

Petra Espinosa.
University of Valladolia, Spain. 15 Oct. - 14 Dec.

Stephan Proennecke.
Paul Scherrer Institute, Villigen, Switzerland. 25 - 31 Mar./21 - 26 Oct.

Ningling Rao.
University of Science and Technology of China, The People's Republic of China. 1 Jan. - 31 Aug.

Ravi Schani.
University of Cambridge, UK. 1 - 14 Jul.

10 Publications

1. **Almdal, K.**; Rosedale, J.H.; Bates, F.S.; Wignall, G.D.; Fredrickson, G.H., Gaussian- to stretched-coil transition in block copolymer melts. Phys. Rev. Lett. (1990) v. 65 p. 1112-1115.

2. **Almdal, K.**; Rosedale, J.H.; Bates, F.S., Order-disorder transition in binary mixtures of nearly symmetric diblock copolymers. Macromolecules (1990) v. 23 p. 4336-4338.

3. **Ananthan, V.S.; Leffers, T.; Hansen, N.**, Cell and band structures in cold-rolled polycrystalline copper. In: Microstructure and Mechanical Processing. Abstracts. Microstructure and Mechanical Processing, University of Cambridge, 28-30 Mar 1990. (The Institute of Metals, London, 1990) p. 4.

4. **Andersen, S.I.; Brøndsted, P.**, Modellering af stål for beregning af cykliske, termiske og mekaniske belastninger. In: Valg af materialer. Dansk Metallurgisk Selskabs vintermøde, Ry, 3-5 Jan 1990. Lilholt, H.; Gundel, P.H. (eds.), (Dansk Metallurgisk Selskab, Lyngby, 1990) p. 13-32.

5. **Bentzen, J.J.; Bilde-Sørensen, J.B.**; Christiansen, N.; **Horsewell, A.**; Ralph, B. (eds.), Structural Ceramics Processing, Microstructure and Properties. 11. Risø International Symposium on Metallurgy and Materials Science, Risø, 3-7 Sep 1990. (Risø National Laboratory, Roskilde, 1990) 539 p.

6. **Bentzen, J.J.**; Schwartzbach, H., Electrical conductivity, structure, and thermal expansion behaviour of ZrO_2-CeO_2-Gd_2O_3-Y_2O_3 solid solutions. Solid States Ionics (1990) v. 40/41 p. 942-946.

7. **Berg, R.; Almdal, K.; Batsberg Pedersen, W.; Holm, A.**; Tam, J.P.; Merrifield, R.B., Polystyrene-grafted polyethylene: Design of film and felt matrices for solid-phase peptide synthesis. In: Innovation and Perspectives in Solid Phase Synthesis. Peptides, Polypeptides and Oligonucleotides. Macro-organic Reagents and Catalysts. 1. International Symposium, Oxford, 29 Aug-2 Sep 1989. Epton, R. (ed.), (SPCC, Birmingham, 1990) p. 453-459.

8. **Berg, R.; Almdal, K.; Batsberg Pedersen, W.; Holm, A.**; Tam, J.P.; Merrifield, R.B., A simple approach to rapid parallel synthesis of multiple peptide analogs. In: Peptides. Chemistry, Structure and Biology. 11. American Peptide Symposium, La Jolla, CA, 9-14 Jul 1989. Rivier, J.E.; Marshall, G.R. (eds.), (ESCOM, Leiden, 1990). p. 1036-1037.

9. **Berg, R.H.; Almdal, K.; Pedersen, W. Batsberg; Holm, A.**, Faste bærematerialer til anvendelse i biosystemer. DK Patent 90559 A (2 Mar 1990).

10. **Berg, R.H.; Almdal, K.; Pedersen, W. Batsberg; Holm, A.**, Tam, J.P.; Merrifield, R.B., Peptide synthesis methods and solid support for use in the method. US Patent 239525 A (1 Sep 1988); WO Patent 89DK201 A (31 Aug 1989); AU Patent 8942258 A (31 Aug 1989).

11. **Berg, R.H.; Almdal, K.; Batsberg Pedersen, W.; Holm, A.**; Tam, J.P.; Merrifield, R.B., Long-chain polystyrene-grafted polyethylene film matrix: A new support for solid-phase peptide synthesis. J. Am. Chem. Soc. (1989) v. 111 p. 8024-8026.

12. **Berg, R.H.; Almdal, K.; Batsberg Pedersen, W.; Holm, A.**; Tam, J.P.; Merrifield, R.B., Peptide synthesis on polystyrene-grafted polyethylene sheets. In: Peptides 1988. Proceedings of the 20. European Peptide Symposium. 20. European Peptide Symposium, Tübingen, 4-9 Sep 1988. Jung, G.; Bayer, E. (eds.), (Walter de Gruyter, Berlin, 1989) p. 196-198.

13. **Berg, R.H.; Almdal, K.; Batsberg Pedersen, W.; Holm, A.**; Tam, J.P.; Merrifield, R.B., Film-supported solid-phase peptide synthesis. In: 21. European Peptide Symposium. Program and abstracts. 21. European Peptide Symposium, Platja d'Aro. Costa Brava, 2-8 Sep 1990. (Universitat de Barcelona, Barcelona, 1990) p. 130.

14. **Borum, K.K.; Gundtoft, H.E.**, Ikke-destruktiv undersøgelse af keramik med ultralyd. Keramisk Information. Nyt fra Centeret for Avanceret Teknisk Keramik (1990) (no.1/2) p. 4-7.

15. Brown, W.; Nicolai, T.; **Hvidt, S.**; Stepanek, P., Relaxation time distributions of entangled polymer solutions from dynamic light scattering and dynamic mechanical measurements. Macromolecules (1990) v. 23 p. 357-359.

16. **Christensen, J.**, Sammenføjning af teknisk keramik. Baggrund og foreløbige resultater. Keramisk Information. Nyt fra Centeret for Avanceret Teknisk Keramik (1990) (no.1/2) p. 7-10.

17. **Clausen, C.**; Holm, P.M., Origin of the acidic volcanics of the Tolfa district, Tuscan Province, Central Italy: An elemental and Sr-isotopic study. Contrib. Mineral. Petrol. (1990) v. 105 p. 403-411.

18. **Debel, C.P.; Andersen, S.I.; Adrian, F.**, Application of the concept of crack-arrest to off-shore structures. Final report on the national research programme: Sprødbrudsarrest i offshore stålkonstruktioner. Risø-Metal-S-8906 (1989) 94 p.

19. **Domanus, J.C.**, Ten years of activities of the Euratom neutron radiography working group. In: Neutron Radiography. Proceedings of the 3. World Conference. 3. World Conference on Neutron Radiography, Osaka, 14-18 May 1989. Fujine, S.; Kanda, K.; Matsumoto G.-I.; Barton, J.P. (eds.), (Kluwer Academic Publishers, Dordrecht, 1990) (EUR-12876) p. 11-18.

20. **Domanus, J.C.**, Practical methods of measuring dimensions from neutron radiographs of nuclear reactor fuel. Nucl. Technol. (1990) v. 92 p. 389-395.

21. El-Sayed Ali, M.; El-Houte, S.; **Sørensen, O.T.**, Preparation and mechanical properties of ceria doped tetragonal zirconia/alumina ceramics. In: Structural Ceramics Processing, Microstructure and Properties. 11. Risø International Symposium on Metallurgy and Materials Science, Risø, 3-7 Sep 1990. **Bentzen, J.J.; Bilde-Sørensen, J.B.**; Christiansen, N.; **Horsewell, A.**; Ralph, B. (eds.), (Risø National Laboratory, Roskilde, 1990) p. 263-268.

22. El-Sayed Ali, M.; El-Houte, S.; **Sørensen, O.T.**, Properties of ceria doped tetragonal zirconia ceramics prepared by coprecipitation technique. In: Structural Ceramics Processing, Microstructure and Properties. 11. Risø International Symposium on Metallurgy and Materials Science, Risø, 3-7 Sep 1990. **Bentzen, J.J.; Bilde-Sørensen, J.B.**; Christiansen, N.; **Horsewell, A.**; Ralph, B. (eds.), (Risø National Laboratory, Roskilde, 1990) p. 269-276.

23. English, C.A.; Green, W.V.; Guinan, M.; **Horsewell, A.**; Ishino, S.; **Singh, B.N.**; Victoria, M., Summary of Silkeborg workshop on radiation damage correlation for fusion conditions. J. Nucl. Mater. (1990) v. 174 p. 352-354.

24. Foreman, A.J.E.; **Singh, B.N.**, Diffusion of helium along grain boundaries during irradiation. Diffus. Defect Data Pt. A (1989) v. 66/69 p. 837-842.

25. Foreman, A.J.E.; **Singh, B.N.**, The role of collision cascades and helium atoms in cavity nucleation. Radiat. Eff. Defects Solids (1990) v. 113 p. 175-194.

26. **Gordes, P.**, Solid oxide fuel cell interconnection materials based on $LaCrO_3$. Risø-M-2867 (1990) 80 p.

27. Green, W.V.; Victoria, M.; **Leffers, T.; Singh, B.N.** (eds.), Proceedings of the workshop on effects of recoil energy spectrum and nuclear transmutations on the evolution of microstructure. Effects of recoil energy spectrum and nuclear transmutations on the evolution of microstructure, Lugano, 24-29 Mar 1988. (Gordon and Breach, New York, 1990) (Radiat. Eff. Defects Solids, 113) *262 p.*

28. **Gundtoft, H.E.; Borum, K.K.**, Ultrasonic non-destructive evaluation of ceramics. In: Structural Ceramics Processing, Microstructure and Properties. 11. Risø International Symposium on Metallurgy and Materials Science, Risø, 3-7 Sep 1990. **Bentzen, J.J.; Bilde-Sørensen, J.B.**; Christiansen, N.; **Horsewell, A.**; Ralph, B. (eds.), (Risø National Laboratory, Roskilde, 1990) p. 319-326.

29. **Hansen, N.**, Deformation microstructures. In: Microstructure and Mechanical Processing. Abstracts. Microstructure and Mechanical Processing, University of Cambridge, 28-30 Mar 1990. (The Institute of Metals, London, 1990) p. 1.

30. **Hansen, N.**, Cold deformation microstructures. Mater. Sci. Technol. (1990) v. 6 p. 1039-1047.

31. **Hansen, N.; Juul Jensen, D.**, Recrystallization of metals containing particles and fibres. In: International Conference on Recrystallization in Metallic Materials. Proceedings. 1. International Conference on Recrystallization in Metallic Materials. Recrystallization '90, Wollongong, 22-26 Jan 1990. Chandra, T. (ed.), (The Minerals, Metals and Materials Society, Warrendale, PA, 1990) p. 79-88.

32. **Horsewell, A.**, Solid sodium particles in aluminium. Phil.Mag. B (1990) v. 62 p. 647-658.

33. **Horsewell, A.; Hansen, N.** (eds.), Metallurgy Department. Annual progress report for 1989. Risø-R-578 (1990) 52 p.

34. **Horsewell, A.** (ed.), Metallurgy Department. Publications 1989. Risø-M-2888 (1990) 44 p.

35. Hughes, D.A.; **Hansen, N.**, The effect of deformation mode on substructural evolution and the formation of shear instabilities. In: Microstructure and Mechanical Processing. Abstracts. Microstructure and Mechanical Processing, University of Cambridge, 28-30 Mar 1990. (The Institute of Metals, London, 1990) p. 3.

36. **Hvidt, S.**; Janmey, P.A., Elasticity and flow properties of actin gels. Makromol. Chem. Macromol. Symp. (1990) v. 39 p. 209-213.

37. **Hvilsted, S.**, A route to quantitative ^{13}C NMR analysis of multicomponent polyesters. In: 9. European Symposium on Polymer Spectroscopy. Book of abstracts. 9. European Symposium on Polymer Spectroscopy, Köln, 25-27 Sep 1990. (Deutsche Forschungsgemeinschaft, Köln, 1990) p. 36.

38. **Hvilsted, S.**; Andruzzi, F.; Paci, M.; Lupinacci, D., Synthesis and characterization of comb-shaped polyesters from 2,2-dioctadecyl-1,3-propanediol and phthalic acids. In: Polycondensation and Related Reactions. Polymers, Properties and Processes. Abstracts. 2. AIM Conference on Advanced Topics in Polymer Science, Gargnano, 10-15 Jun 1990. Berti, C.; Pilati, F. (ed.), (Associazione Italiana di Scienza e Tecnologia delle Macromolecole, Gargnano, 1990) p. 67.

39. **Hvilsted, S.**; Andruzzi, F.; Paci, M., Synthesis and characterization of comb-shaped polyesters from 2-octadecyl- and 2,2-dioctadecyl-1,3-propanediol and diphenyl esters. In: 33. IUPAC International Symposium on Macromolecules. Book of abstracts. 33. IUPAC International Symposium on Macromolecules, Montreal, 8-13 Jul 1990. (IUPAC, Montreal, 1990) Session 1.9.4.

40. Janmey, P.A.; **Hvidt, S.**; Oster, G.F.; Lamb, J.; Stossel, T.P.; Hartwig, J.H., Effect of ATP on actin filament stiffness. Nature (1990) v. 347 p. 95-99.

41. Janmey, P.A.; **Hvidt, S.**; Lamb, J.; Stossel, T.P., Resemblance of actin-binding protein/actin gels to covalently crosslinked networks. Nature (1990) v. 345 p. 89-92.

42. **Jensen, K.O.**; **Eldrup, M.**; Linderoth, S.; Evans, J.H., Influence of krypton physisorption on the positron surface state lifetime: Rates of positron trapping into cavities. J. Phys. Condens. Matter (1990) v. 2 p. 2081-2091.

43. **Juul Jensen, D.**; **Hansen, N.**; **Liu, Y.L.**, Effect of recrystallization temperature on texture and grain size of an Al-SiC composite. In: Microstructure and Mechanical Processing. Abstracts. Microstructure and Mechanical Processing, University of Cambridge, 28-30 Mar 1990. (The Institute of Metals, London, 1990) p. 73.

44. **Juul Jensen, D.**, Texture transformation during annealing. In-situ measurements and computer modelling. Nondestr. Test. Eval. (1990) v. 5 p. 335-347.

45. **Juul Jensen, D.**; **Hansen, N.**, Modelling of recrystallization texture and microstructure. In: International Conference on Recrystallization in Metallic Materials. Proceedings. 1. International Conference on Recrystallization in Metallic Materials. Recrystallization '90, Wollongong, 22-26 Jan 1990. Chandra, T. (ed.), (The Minerals, Metals and Materials Society, Warrendale, PA, 1990) p. 661-666.

46. **Juul Jensen, D.**; Schmidt, N.H., An automatic on-line technique for determination of crystallographic orientations by EBSP. In: International Conference on Recrystallization in Metallic Materials. Proceedings. 1. International Conference on Recrystallization in Metallic Materials. Recrystallization '90, Wollongong, 22-26 Jan 1990. Chandra, T. (ed.), (The Minerals, Metals and Materials Society, Warrendale, PA, 1990) p. 219-224.

47. **Juul Jensen, D.**; **Hansen, N.**, Flow stress anisotropy in aluminium. Acta Metall. Mater. (1990) v. 38 p. 1369-1380.

48. **Kindl, B.**; **Hansen, N.**; **Sørensen, O.T.**, Ceramic manufacture with inorganic processing aids. In: Structural Ceramics Processing, Microstructure and Properties. 11. Risø International Symposium on Metallurgy and Materials Science, Risø, 3-7 Sep 1990. **Bentzen, J.J.**; **Bilde-Sørensen, J.B.**; Christiansen, N.; **Horsewell, A.**; Ralph, B. (eds.), (Risø National Laboratory, Roskilde, 1990) p. 371-376.

49. **Kindl, B.**; **Liu, Y.-L.**; **Hansen, N.**, Fremstilling af materialer. DK Patent 893366 A (6 Jul 1989).

50. **Kindl, B.**; **Mogensen, M.**; **Poulsen, F.W.**, First Danish solid oxide fuel cell tests. In: SCAN-ELCHEM-90. Abstracts. Open Scandinavian Symposium on New Developments in Fundamental and Applied Electrochemistry, Copenhagen, 7-10 Oct 1990. Hammerich, O.; Jensen, O.J.; Ulstrup, J. (eds.), (Danish Electrochemical Society, Copenhagen, 1990) SC03.

51. **Leffers, T.**; **Bilde-Sørensen, J.B.**, Intra- and intergranular heterogeneities in the plastic deformation of brass during rolling. Acta Metall. Mater. (1990) v. 38 p. 1917-1926.

52. **Leffers, T.**; **Juul Jensen, D.**, The effect of bending and straightening on rolling texture and microstructure in brass. Scr. Metall. Mater. (1990) v. 24 p. 2431-2435.

53. **Lilholt, H.**, Bergsøe-medaljen tildelt Michael F. Ashby. In: Valg af materialer. Dansk Metallurgisk Selskabs vintermøde, Ry, 3-5 Jan 1990. **Lilholt, H.**; Gundel, P.H. (eds.), (Dansk Metallurgisk Selskab, Lyngby, 1990) p. 443-445.

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Abstract (Max. 2000 characters)

Selected activities of the Materials Department at Risø National Laboratory during 1990 are described. The work is presented in three chapters: *Materials Science, Materials Engineering* and *Materials Technology*. A survey is given of the Department's participation in international collaboration and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists, publications, lectures and poster presentations are included.

Descriptors INIS/EDB

CERAMICS;COMPOSITE MATERIALS; ENERGY STORAGE SYSTEMS; FUEL CELLS; MATERIALS TESTING; PHYSICAL METALLURGY; POLYMERS; PROGRESS REPORT; RESEARCH PROGRAMS; RISOE NATIONAL LABORATORY

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Risø National Laboratory is a broad-based research organization with primary research activities in energy, the environment and in materials. There is a total of 957 employees.

The Materials Department* employs 43 scientists and engineers and 54 technical and office staff. The research programmes include basic studies of materials structure and properties, structural mechanics and materials testing, and processing techniques for polymers, polymer composites, powder metallurgical products and engineering ceramics. Advanced characterization techniques used in the Department are electron microscopy, positron annihilation, neutron diffraction, small angle neutron scattering, Fourier-transform infra red spectroscopy and mechanical testing.

The activities of the Materials Department are supported by a combination of basic government funding, national, EEC and international project funded research and through industrial contracts.

Within the Danish Programme of Materials Technology, the Materials Department participates in the Centres of Polymer Composites, Advanced Technical Ceramics and Powder Metallurgy.

*The Department, previously called the »Metallurgy Department«, changed its name in May 1990.

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